ANALYTICAL ABSTRACTS

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9. **Further observations on automatic titrimetry.** J. Haslam and D. C. M. Squirrell (I.C.I. Ltd., Plastics Div., Welwyn Garden City, England). *Analyst*, 1957, **82**, 511–517.—Since the previous report (*Analyst*, 1954, **79**, 689), additional experience of automatic titrimetry has been gained. Details are now given of its application to the titration of iron and aluminium with EDTA, the determination of water in organic compounds by the Karl Fischer method, the determination of formaldehyde by the neutral sulphite method, the detection of Terylene by aminolysis, the determination of nylon 610 salt in moist material, and the determination of aldehydes and ketones in methyl methacrylate monomer. Observations are made on the determination of nitrogen and chlorine in polymers.
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R. Přibil (Res. Inst. Pharm. and Biochem., Prague, Czechoslovakia). *Chem. Listy*, 1957, **51** (6), 1061–1067.—Methylthymol blue (**I**) (3;3′-bis-NN-di(carboxymethyl)aminomethylthymolsulphonephthalein) reacts in acid, as well as in alkaline, medium with several cations, yielding intense blue chelates. The complexometric determinations of Bi, Th, Sc, La, Pb, Zn, Cd, Hg and Co in acid medium, and the determination of Pb, Zn, Cd, Co, Mn, Mg, Ca, Sr and Ba in alkaline (aq. NH_3 or NaOH) medium, with the use of **I** as indicator, are described. The following media were used for the titrations—*acid soln.*: *N* HNO_3 (Bi), acetate buffer soln. pH 3 (Th), pyridine (Sc), hexamine (La, Pb, Zn, Cd, Co, Hg); *alkaline soln.*: aq. NH_3 (Zn, Co, Ca, Sr, Ba), aq. NH_3 - K Na tartrate (Pb), aq. NH_3 - buffer soln. of aq. NH_3 - NH_4Cl (pH 10) (Cd, Mg), aq. NH_3 - hydroxylamine hydrochloride (Mn), 0·05 *N* NaOH (Ca, Sr, Ba). A 0·1% soln. of **I** in H_2O, or a mixture of **I** with KNO_3 (1:100) was used; the colour change was from blue to yellow (acid medium) or grey to blue (alkaline medium). In all the titrations described a vol. of about 50 to 60 ml and 0·05 *M* EDTA (disodium salt) were used. J. ZÝKA

11. **Practical applications of gas chromatography.** W. Leithe. *Öst. ChemZtg*, 1957, **58** (11–12), 141–148.—This general review with 45 references lays special emphasis on gas - liquid partition chromatography. E. G. CUMMINS

12. **Use of ion-exchange resins with non-aqueous and mixed solvents.** O. D. Bonner (Univ. of S. Carolina, Columbia, U.S.A.). *J. Chem. Educ.*, 1957, **34**, 174–177.—A review.

13. **Improved technique for electrophoresis in agar gel.** C. Wunderly and V. Bustamente (Med. Univ.-Klinik, Zürich, Switzerland). *Klin. Wochschr.*, 1957, **35** (15), 758–760.—An agar film is made on a glass plate and the serum applied by contact of a strip of filter-paper saturated with the specimen. After electrophoresis, the film is stained and then evaluated directly in the Elphor apparatus. H. F. W. KIRKPATRICK

14. **Photometric evaluation of non-transparent paper electropherograms.** H. Remky (Augenklinik, Univ. München, Germany). *Klin. Wochschr.*, 1957, **35** (12), 635–636.—A mathematical relationship exists between the extinction values obtained for electropherograms before and after making them transparent. The construction of a nomogram to correct readings given by non-transparent strips is described. H. F. W. KIRKPATRICK

15. **Report of the fifth seminar of the Centro Ricerche Spectrochimiche, Gazzada (Varese), of the Associazione Italiana di Metallurgia.** *Metallurg. Ital.*, 1957, **49** (6), 397–487.—The following papers are included (in Italian unless otherwise stated). **Principles, methods and apparatus of emission spectrographic analysis,** J. Junkes, 399–404. **The development of spectrographic analysis in the laboratories of the Italian metallurgical industry in the last decade and the activity of the C.R.S.,** O. Masi, 405–408. **Some particular applications of emission spectrography,** E. Loeuille, 409–410 (in French). **Some considerations on the selection of metallic specimens for spectrographic analysis,** H. Guyer, 411–414 (in French). **Normal and anomalous inhibitions and sensitised emissions,** M. Pierucci, 415–418. **'Enhancements' and 'attenuations' in spectrochemical analysis. Study in relation to energy of source and to excitation potentials of the lines of the elements. Analytical deductions,** V. Gazzi, 419–424. **Photographic photometry,** J. Junkes and E. W. Salpeter, 425–430. **Effective line width as a measure of intensity,** R. Intonti, H. Pfundt and A. Lais, 431–443. **Blackening and effective line width,** R. Passeri, 444–448. **The 'CF4 Optica' spectrophotometer,** B. Polli, 462–464. **Determination of sodium in aluminium oxide and hydroxide and in 'red mud' (Bayer process) by flame spectrophotometry with the Beckman spectrophotometer,** L. Braicovich and M. F. Lani, 465–474. **New equipment for direct-reading analysis,** H. Bückert and I. Raffaele, 475–478. **Comparison between successive-integration and parallel-integration quantometers for spectrochemical analysis,** M. Scalise, 479–482. **The complementary nature of X-ray fluorescence and optical emission methods in spectrochemical analysis,** M. Scalise, 483–487. BULL. B.N.F.M.R.A.

16. **Dependence of background associated with high-voltage a.c. arc spectra on certain parameters.** A. Strasheim (Nat. Phys. Res. Lab., Pretoria, S. Africa). *Appl. Spectroscopy*, 1957, **11** (2), 76–83.—The experiment described seems to indicate that the line spectra of analysis elements and the associated background are excited concurrently in an a.c. arc. The intense OH band spectra between 3063 and 3300 A can be resolved by operating the arc in an oxygen atmosphere and this results in more accurate line-intensity ratios. Carbon dioxide is unsatisfactory. Of the arc parameters, namely electrode separation and arc current, the arc current is the more critical and must be regulated to 0·1 amp. K. A. PROCTOR

17. **Quantitative ultra-violet vapour spectrophotometry.** R. A. Friedel (Bureau of Mines, Bruceton, Pa., U.S.A.). *Appl. Spectroscopy*, 1957, **11** (2), 61–64.—Some applications of u.v. vapour spectrophotometry, including quant. analyses, are discussed and a simple technique is described for obtaining quant. vapour spectra of normally liquid compounds. Deviations from Beer's law in the sharp bands of benzene and toluene vapours have been studied; they may be due to finite slit widths or to nearby scattered radiation. K. A. PROCTOR

18. **Developments in the polarographic analysis of inorganic compounds.** I. V. Pyatnitskiĭ. *Zavod. Lab.*, 1957, **23** (6), 668–678.—A review is presented of work carried out in 1954 to 1955. (170 references.) G. S. SMITH

19. **Neutron activation cross-section graphs.** W. W. Neinke and R. S. Maddock (Dept. Chem., Univ. Michigan, Ann Arbor, U.S.A.). *Anal. Chem.*, 1957, **29** (8), 1171–1174.—Revised values of all known thermal neutron activation cross-sections have been plotted against the half-life of the radioisotope produced. The standard error of each value is also included. K. A. PROCTOR

2.—INORGANIC ANALYSIS

20. **Titrimetry in the study of inorganic reactions in non-aqueous solvents.** O. W. Kolling (Friends Univ., Wichita, Kansas, U.S.A.). *J. Chem. Educ.*, 1957, **34**, 170–173.—A survey is presented of the literature on titration of inorg. compounds in

solvents such as acetic acid, acid chlorides, aliphatic amines, $AsCl_3$, hydrocarbons, chlorinated hydrocarbons, alcohols, liquid NH_3 and liquid SO_2. (49 references.) S.C.I. ABSTR.

21. **Determination of metals by differential polarimetry.** M. Freegarde (Bragg Lab., N.O.I.D., Janson St., Sheffield). *Chem. & Ind.*, 1957, (28), 984–985.—At a const. temp., wavelength and length of polarimeter tube, the optical rotation of an active solution is a linear function of concn. This is the basis of the new technique described. The optical rotation of a water-soluble, optically active reagent, which reacts quant. with the metal to be determined, is measured. Then a soln. of the metal is added to an excess aliquot of the reagent soln., and the optical rotation of the system formed is a measure of the quantity of metal added. Tests with Ag^+ and Fe^{3+}, with D-tartaric acid as reagent, gave, in both cases, a linear relationship between optical rotation and quantity of metal added, although sensitivity was low. Methods for increasing the sensitivity are suggested. L. S. ADLER

22. **Analysis of rare gases by mass spectrometer.** V. Čermák (Inst. Phys. Chem. Acad. Sci., Prague, Czechoslovakia). *Chem. Průmysl*, 1957, **7** (1), 8–11.—Results obtained by the use of the mass spectrometer in works control in the chemical industry are reviewed. The smallest detectable amounts are—He $2{\cdot}5 \times 10^{-3}$, Ne $1{\cdot}5 \times 10^{-3}$, A 3×10^{-4}, Kr 4×10^{-4}, and Xe 6×10^{-4}, per cent. by vol. J. BÖSWART

23. **Photometric determination of hydrogen peroxide.** J. Čelechovský, E. Krejčí and V. Krejčí (Masaryk Univ., Brno, Czechoslovakia). *Českosl. Farm.*, 1957, **6** (2), 103–105.—A stable coloured complex is formed by the oxidation of Fe^{2+} with H_2O_2 in the presence of salicylic (**I**) or sulphosalicylic acid (**II**). The extinction is measured between 490 and 533 mμ. Soln. of pH 2 to 2·2, 8×10^{-3} *M* Fe^{2+} and 0·1 *M* **I** or **II** are recommended. Concn. of 18 to 260 μg of H_2O_2 in 50 ml can be reliably determined. Reducing or oxidising agents interfere. The method was successful in the determination of metal peroxides, *e.g.*, MgO_2. J. VOLKE

24. **Determination of traces of lithium, sodium, potassium and calcium by use of a simple flame photometer.** H. Baranska (Dept. Tech. Physics, Inst. Gen. Chem., Warsaw). *Chem. Anal., Warsaw*, 1957, **2** (2), 138–148.—Traces of Li, Na, K and Ca occurring together are determined by a rapid flame-photometric method. The time for a single determination is 30 sec. The method is applicable to the following concn.—Li, 0·1 to 10 p.p.m.; Na, 1 to 100 p.p.m.; K, 0·5 to 100 p.p.m.; Ca, 5 to 100 p.p.m. It is found that at these concn. the mutual interference is negligible and does not exceed the experimental error. To eliminate errors due to interference, parallel analyses are made on mixtures of similar composition. Interference due to anions, *e.g.*, Br^-, Cl^- and NO_3^-, present up to 10 p.p.m. is negligible. H. DMOWSKA

25. **Application of the method of differences to the determination of sodium with a flame photometer.** J. Dobrowolski and N. Wyszyński (Dept. of General Chem., Pomeranian Med. Acad., Szczecin, Poland). *Chem. Anal., Warsaw*, 1957, **2** (1), 11–21.—On different occasions, the absolute values for the deflections of the galvanometer of a flame photometer differ for the same concn. of Na, but the differences of these values for a series of concn. are nearly constant. To plot a calibration curve, several series of measurements are made on standard Na soln. (10 to 100 μg of Na per ml) and the mean differences of the galvanometer readings for each concn. of Na are calculated. When a sample soln. of Na is analysed, a standard soln. (concn. $\simeq 25$ μg of Na per ml) is also tested, and the appropriate differences are added to or subtracted from the reading obtained to give a series covering the concn. range for the calibration curve. The Na content of the sample soln. is read from the new curve. The relative error for concn. < 50 μg of Na per ml did not exceed 0·5%. The method may also be applied to the flame-photometric determination of other metals. K. F. SPOREK

26. **Indirect complexometric titration of sodium and potassium with EDTA.** Buddhadev Sen (State Univ., Baton Rouge, La., U.S.A.). *Z. anal. Chem.*, 1957, **157** (1), 2–6 (in English).—The Na in 2 ml of the sample soln. is pptd. as sodium zinc uranyl acetate, which is washed and dissolved in hot H_2O. After the addition of ammonium carbonate (1 g) and ammoniacal buffer (3 ml), the Zn in the soln. (50 to 75 ml) is titrated with 0·01 *M* EDTA (disodium salt), with Eriochrome black T as indicator. The K in 10 ml of the sample soln. is pptd. as potassium sodium cobaltinitrite, which is dissolved in hot H_2O containing 1·5 ml of 2 *N* HCl. The cool soln. is made neutral with aq. NH_3, 1 g of NH_4SCN and an equal vol. of acetone are added, and the blue soln. is titrated with 0·01 *M* EDTA until colourless. The accuracy is equal to that of the corresponding gravimetric methods, and any interfering ions should be controlled as in those methods. W. J. BAKER

27. **Volumetric determination of potassium.** E. D. Schall (Purdue Univ., Lafayette, Ind., U.S.A.). *Anal. Chem.*, 1957, **29** (7), 1044–1046.—The sample (2·5 g), *e.g.*, fertiliser, is boiled with excess of ammonium oxalate and water, and the soln. is made alkaline with aq. NH_3. The mixture is made up to standard vol. and an aliquot of the supernatant liquid is treated with aq. NaOH and formaldehyde and then with an excess of Na tetraphenylboron soln. The soln. is filtered and an aliquot of the filtrate is titrated with standard cetyltrimethylammonium bromide, with bromophenol blue as indicator. The average error is given as 0·3% up to 100 mg of K. A comparison with results obtained with the gravimetric procedure shows the volumetric figures to be slightly low. Interference is caused by Cs^+, Rb^+, Ag^+, Hg^{2+} and N bases. D. A. PANTONY

28. **Complexometric determination of potassium.** R. Pilleri (Univ., Turin, Italy). *Z. anal. Chem.*, 1957, **157** (1), 1–2.—To 10 ml of the sample soln. ($\equiv$ 0·02 to 0·05 g of K) are added 3 ml of ethanol, 1 ml of aq. 7% KNO_3 and 5 ml of aq. 20% (w/v) sodium cobaltinitrite soln. The mixture is warmed for $\simeq$15 min. on the water bath and, after 3 to 4 hr., the ppt. of $K_2Na[Co(NO_2)_6].H_2O$ is collected, washed, and dissolved in hot conc. H_2SO_4. The diluted soln. is made just neutral with aq. NH_3, 20 ml of aq. 0·1 *M* EDTA (disodium salt) and 10 ml of ammoniacal buffer (pH 10) are added, and the excess of EDTA is then titrated with aq. 0·1 *M* $ZnSO_4$, with Eriochrome black T indicator. W. J. BAKER

29. **Comparative characteristics of some analytical methods of determining potassium as applied to silicates.** G. A. Murina, A. D. Iskanderova, V. D. Sprintssov. *Inform. Sb. Vses Nauch. Geol. Inst.*, 1956, (3), 134–137; *Ref. Zhur., Khim.*, 1957, Abstr. No. 27,140.—With the help of the radioactive indicators (^{42}K and ^{24}Na) it has been shown that the perchlorate and dipicrylamine methods of determining K are no less accurate than the chloroplatinate method. The rapid dipicrylamine method as described is suitable for the determination of K in various silicate rocks and is considerably less laborious than the chloroplatinate method.

L. Herdan

30. **Polarographic estimation of univalent copper in the presence of bivalent copper in ammoniacal solution of cuprous acetate.** J. Maďar (Výskumný ústav ropy, Bratislava, Czechoslovakia). *Chem. Průmysl*, 1957, **7** (6), 299–300.—The use of polarography enables the simultaneous determination of Cu^{I} and Cu^{II} to be carried out. The procedure and a comparison of results are given.

J. Böswart

31. **The determination of colouring matter in glass by surface treatment. (Determination of copper.)** L. I. Ganago and N. A. Tananaev. *Trudy Ural'sk. Politekh. Inst.*, 1956, (57), 126–128; *Ref. Zhur., Khim.*, 1957, Abstr. No. 30,965.—Place two drops of HF soln. in a wax-ringed area of the surface of the cleaned sample and of standard glasses. After 5 min. add 3 or 4 drops of water, transfer the soln. to a container, dilute to 1·5 or 2 ml, add 3 or 4 drops of H_2SO_4 (1 : 1) and boil to obtain a clear soln. Add 1 or 2 ml of water, 6 to 8 drops of a saturated pyrophosphate soln. and 10 to 15 drops of a 0·1% soln. of Na diethyldithiocarbamate. Compare the colours formed. C. D. Kopkin

32. **Analysis of copper and lead slags.** S. Yu. Faĭnberg, A. A. Blyakhman and S. M. Stankova (State Sci. Res. Inst. of Non-ferrous Metals). *Zavod. Lab.*, 1957, **23** (6), 647–652.—The analysis of slags containing high percentages of SiO_2 and Fe for Al_2O_3 (≃2 to 5%), CaO (≃5 to 8%), MgO (≃0·4 to 0·8%) and SiO_2 is described. The methods are suitable for use in the presence of Cu and Pb. To determine Al_2O_3, the sample (0·5 g) is fused with previously melted and cooled NaOH (3 g) to which is added Na_2O_2 (2 g). The aq. soln. of the melt is treated with conc. HCl (20 ml) and an aliquot of the soln. is treated with NH_4Cl and aq. NH_3 to precipitate $Al(OH)_3$ and $Fe(OH)_3$, which are separated and dissolved in 2×20 to 25 ml of dil. HCl (1 + 1). The Fe is extracted by means of amyl acetate, and the aq. layer with the addition of 30% H_2O_2 (2 drops) is evaporated to dryness, dil. HCl is added to the residue and the evaporation is repeated, after which the residue is dissolved in water (20 ml) containing HCl. A suitable aliquot in a 100-ml flask is mixed with 0·5 ml of a freshly prepared 2·2% soln. of ascorbic acid, 30 ml of an acetate buffer soln. (pH 5·4) and 15 ml of 0·02% stilbazo soln. The soln. is diluted to the mark and the extinction is measured after 15 min. on a photocolorimeter with a green filter and a 50-mm cell. To determine MgO, Titan yellow is used after all interfering elements have been removed from the solution of a melt obtained as described above. Silica is determined by the molybdenum blue method with $SnCl_2$ as reducing agent. To determine CaO, an aliquot (100 ml) of the acid-treated soln. (500 ml) of the melt as obtained in the method for Al_2O_3 is treated with powdered ammonium oxalate (1 g), 5 or 6 drops of methyl red indicator, and then with aq. NH_3 (1 + 1) until the soln. is yellow. A further 0·5 g of ammonium oxalate is then added, the soln. is boiled and kept for 30 min. at 70° to 80° and filtered. If the whole of the ammonium oxalate (1·5 g) is added initially, the neutralisation endpoint with aq. NH_3 is less definite. The ppt. is washed as usual and dissolved in dil. H_2SO_4, and the soln. is titrated with 0·05 *N* $KMnO_4$.

G. S. Smith

33. **Polarographic determination of copper and bismuth in tartrate solution.** I. V. Pyatnitskiĭ and A. P. Kostyshina. *Ukr. Khim. Zhur.*, 1956, **22** (5), 679–686; *Ref. Zhur., Khim.*, 1957, Abstr. No. 30,935.—The composition and stability of the tartrate complexes of Bi and Cu were studied potentiometrically. The half-wave potentials of Bi and Cu in tartrate soln. were calculated. The separate polarographic determination of Bi and Cu is possible when both are present in a basal soln. of 0·4 *M* tartrate at a pH of 3·5 to 5·5. C. D. Kopkin

34. **The use of catalysis-inhibition (anti-catalysis) in analytical chemistry. The determination of silver and mercury with the ceric sulphate - arsenous acid - sodium iodide system. (Preliminary communication.)** T. Braun (Inst. Med. Sci. and Pharm., Marosvásárhely, Romania). *Magyar Kém. Foly.*, 1957, **63** (1), 39–40.—The catalytic determination of traces of I^- by means of the $As(OH)_3$ - $Ce(SO_4)_2$ reaction is strongly inhibited by traces of Ag and Hg. The anti-catalytic effect is proportional to the amount of inhibitor, if the amount of I^- is constant. *Procedure*—Place drops of the soln., each containing 0·5 μg of I^-, on the lower ends of six strips of chromatographic paper. Dry them in air, then on the same spots place standard soln. containing an increasing amount (0·005 to 0·05 μg) of Ag or Hg. Dry and suspend the strips in the chromatographic cabinet, with their lower ends dipping into the developing soln. This soln. consists of a mixture (1 : 1 : 2·5) of $Ce(SO_4)_2$ - $2(NH_4)_2SO_4$ (10 g) dissolved in warm *N* H_2SO_4 (100 ml) and filtered, As_2O_3 (5 g) in 30% NaOH soln. (7 ml) diluted to 100 ml with *N* H_2SO_4 (freshly prepared before use) and *N* H_2SO_4. On drying the chromatogram, and especially in u.v. light, colourless zones of various lengths appear. Their length is proportional to the amount of the catalysis inhibitor. A. G. Peto

35. **Determination of gold in sea water by radioactivation analysis.** R. W. Hummel (Anal. Chem. Group, A.E.R.E., Harwell, England). *Analyst*, 1957, **82**, 483–488.—The samples of sea water in silica tubes were irradiated in the Harwell pile together with standard soln. of gold and blank tubes. The analytical procedure described is based on that of Goldberg *et al.* (*Brit. Abstr. C*, 1950, 314). The irradiated gold with added inactive gold carrier was extracted from a soln. in HCl with ethyl acetate, the solvent was removed and the residue dissolved in aqua regia; most of the HNO_3 was removed and the gold was pptd. from HCl soln. with quinol. It was then weighed, its activity counted, corrected and compared with that of the standard soln. A. O. Jones

36. **Analytical precipitation of gold from gold-plating electrolytes with hydrazine hydrochloride.** Z. P. Zharkova and E. I. Zhacheva. *Nauch.-Issled. Tr. Tsentr. Nauch. Inst. Vspomogat. Izdeliĭ*

i Zapas. Detaleĭ k Tekstil'n. Oborud., 1956, (4), 44–47; *Ref. Zhur., Khim.*, 1957, Abstr. No. 30,966.—A method for the determination of Au in cyanide extracts of ores is modified for application to Au in gold-plating electrolytes. Neutralise the sample of electrolyte (containing 0·005 to 0·05 g of Au) with HCl to phenolphthalein, heat to boiling-point, add a 10% soln. of hydrazine hydrochloride (50 ml) and boil, maintaining the vol. of the soln. Filter off the ppt. of Au, wash, dry, and ignite at 800°. To the filtrate add 25 ml of the hydrazine soln. and boil, to ensure complete pptn. Dissolve the ignited ppt. in aqua regia (5 ml), evaporate to dryness and evaporate the residue three times with 1 or 2 ml of dil. HCl (1:3). Add dil. HCl (1:50) (3 ml) and two or three drops of a HCl soln. of Br, evaporate, dilute to 30 or 40 ml, add KHF_2 (0·1 g) and two or three drops of *o*-dianisidine (10 mg in 10 ml of HCl, 1:300) and after 5 min. titrate with a soln. of quinol till colourless or pale yellow.

C. D. KOPKIN

37. **Quantitative separation of beryllium from certain elements by ion-exchange chromatography.** T. A. Belyavskaya and V. I. Fadeeva. *Vestn. Moskov. Univ.*, 1956, (6), 73–79; *Ref. Zhur., Khim.*, 1957, Abstr. No. 30,967.—The conditions are studied for the adsorption of Be on the ion-exchange resin SBS, its dependence on pH, and desorption with HCl, alkalis, and soln. of ammonium carbonate and $NaHCO_3$. A method is described for the quant. separation of Be from Cu and Ni. To a soln. containing Be and Cu or Ni add aq. NH_3 and excess of a 10% soln. of ammonium carbonate (pH 8·5 to 9). Pass the soln. through a column of SBS (NH_4^+ form) (height 20 cm, diam. 0·7 cm) at a rate of 1·5 to 2 ml per min. Wash the column with the ammonium carbonate soln. (20 to 30 ml), combine the soln. and, after boiling out CO_2, determine Be. The desorption of the Cu or Ni is carried out with 2 *N* HCl. The method gives satisfactory results with ratios of Be to Cu or Ni of $\simeq$1:1 to 1:100. A method is also described for the separation of Be from MoO_4^{2-} and PO_4^{3-}. Pass the soln. (pH 3 to 4, 20 to 30 ml) through a column of SBS (NH_4^+ form) at a rate of 1 to 1·5 ml per min. Wash the column with 25 ml of 5% aq. NH_3 and 25 ml of water. Combine the liquors and determine Mo or P. The Be may be desorbed with 2 *N* HCl.

C. D. KOPKIN

38. **Standards for the spectrographic analysis of magnesium, its alloys and some aluminium alloys.** L. R. Pittwell (Dominion Magnesium Ltd., Haley, Ontario, Canada). *Appl. Spectroscopy*, 1957, **11** (2), 67–73.—A method for preparing the standards in the form of pencils is described, together with methods for analysing them for homogeneity and composition.

K. A. PROCTOR

39. **Direct complexometric determination of magnesium in aluminium alloys with Eriochrome black.** T. L. Beránek (Res. Inst., ČKD-Stalingrad, Prague, Czechoslovakia). *Hutn. List.*, 1957, **12** (5), 434–435.—Interfering elements such as Al, Cu, Zn, Fe, Ni and Mn are masked with triethanolamine and KCN in a reducing medium of hydroxylamine at pH 10. The titration is carried out with EDTA (disodium salt) (**I**), with Eriochrome black T as indicator. *Procedure*—Dissolve the sample (1 g) in dil. HCl (1:1) (20 ml), evaporate, and dissolve the residue in water (200 ml). To a 20-ml aliquot add water (80 ml) and hydroxylamine hydrochloride and cool to about 15°. Add triethanolamine (10 ml), NH_4Cl - aq. NH_3 buffer soln. (10 ml), KCN soln. (10%) (10 ml) and Eriochrome black T. Titrate with 0·01 *M* **I**.

J. BÖSWART

40. **Direct chelatometric method for the estimation of calcium or magnesium in the presence of phosphate.** M. R. Verma, V. M. Bhuchar and K. J. Therattil (Nat. Phys. Lab. of India, New Delhi). *Nature*, 1957, **179**, 1244.—Equivalent quantities of a soln. of zinc and the disodium salt of 1:2-diamino-*cyclo*hexane-NNN′N′-tetra-acetic acid (**I**) are mixed. The resulting soln. is mixed with an aliquot of the soln. of the calcium or magnesium phosphate to be determined and the pH is adjusted to between 3 and 4. After being shaken, the soln. is buffered to a pH of 8 to 10 and titrated against a soln. of **I**, with Eriochrome black T as indicator. Typical results gave a mean error of +0·2% for CaO from four determinations (max. +0·6%, min. −0·1%). For MgO, three determinations gave errors of −0·2, +0·3 and zero, respectively.

L. S. ADLER

41. **Rapid method for the determination of calcium and magnesium in limestone and dolomite with Complexone III [EDTA].** J. Dereń, J. Haber and J. Nedoma (Dept. of Inorg. Chem., Mining-Metallurgic Acad., Kraków). *Chem. Anal., Warsaw*, 1957, **2** (1), 3–10.—EDTA (disodium salt) is used for titrating Ca^{2+} in dil. NaOH soln., with murexide as indicator, and total Ca^{2+} and Mg^{2+} in aq. NH_3 - NH_4Cl buffered soln. in the presence of Eriochrome black T indicator; Mg is calculated by difference. The method can be applied to samples of limestone without the separation of other ions. The results agreed well with those given by the oxalate and the phosphate methods for the determination of Ca and Mg, respectively.

K. F. SPOREK

42. **Colorimetric determination of small amounts of calcium.** G. Stănescu. *Rev. Chim., Bucharest*, 1957, **8** (4), 260–261.—The usual method of estimating small amounts of Ca by pptn. as oxalate, dissolution of the ppt. in HCl and colorimetric estimation of oxalic acid by decolorisation of $Fe(SCN)_3$ is modified—the Ca is pptd. by titration with K oxalate, the ppt. is centrifuged off, and the excess of oxalate is estimated colorimetrically. *Procedure*—To 2 ml of a boiling soln., weakly alkaline or neutral (pH = 7·0 to 7·8), containing between 0·1 and 1·0 mg of Ca, is added drop by drop an excess of K oxalate soln. After being set aside for 10 min., the product is boiled for 15 min., cooled and made up to 50 ml. A portion of this is centrifuged for 20 to 30 min. at 5000 r.p.m., and a 5 to 10-ml aliquot is mixed with 10 ml of $Fe(SCN)_3$ soln. After 15 min. the colour is measured in a Pulfrich apparatus. The colour is inversely proportional to the amount of oxalate. Results can be estimated by reference to a standard curve. Errors are within 5% of the admitted error of the original colorimetric method.

H. SHER

43. **Rapid semi-micro cation-exchange method for determining calcium in cement.** V. N. Lenskaya and L. M. Kul'berg. *Uch. Zap. Saratovsk. Univ.*, 1956, **43**, 141–144; *Ref. Zhur., Khim.*, 1957, Abstr. No. 30,972.—Treat the sample of cement (20 to 30 mg) with conc. HCl (0·5 ml), dilute to 10 ml with water and pass the soln. through a cationite (PFSK, H form). Wash the column with 10 ml of water, then pass hot 0·1 *N* ammonium oxalate (100 ml) through the column. Acidify the eluate, containing Fe, Al, Mg, alkali metals and the excess of $C_2O_4^{2-}$, with dil. H_2SO_4 (1:5, 25 ml), heat to 70° or 80°

and titrate the $C_2O_4^{2-}$ with 0·1 *N* $KMnO_4$. The removal of SiO_2 and R_2O_3 from the cement is unnecessary. C. D. KOPKIN

44. **Purity tests using flame photometry. Sodium and potassium as contaminants in calcium salts.** H. Aasness (Statens Farmakopélab., Blindern). *Medd. Norsk Farm. Selsk.*, 1957, **19** (4), 53–63.—For the determination of Na in $CaCl_2$ the apparatus employed was that of Brealey and Ross (*Analyst*, 1951, **76**, 334) with the addition of a wavelength scanning device and recorder, but for K a filter photometer was used; with this apparatus "flame pure" substances should be used for preparing standard soln. *Procedure*—For the determination of Na with a direct-reading monochromator, dissolve 2·5 g of dried or 5 g of hydrated $CaCl_2$ in water and make up to 100 ml. The flame photometer is adjusted so that the reading at 5890 A is 100 for a soln. containing 10 p.p.m. of Na and the same amount of Ca as in the test substance. The standard curve is constructed with soln. containing 0·1, 3, 5, 8 and 10 p.p.m. of Na and of Ca. Readings are made on the galvanometer at the max. of about 5890 A and at the minimum of approx. 5920 A, the difference between these readings being a measure of the Na content. For the determination of K, a similar procedure is adopted. Readings are made, with a suitable filter, or at 7680 A. If the readings for the test soln. are >100 the soln. must be diluted tenfold and readings must be redetermined. It is usual to make up standard soln. containing the same amount of interfering material as is present in the test soln. P. HAAS

45. **Determination of traces of lithium in calcium hydroxide by spectrographic analysis.** S. Skalska and S. Held (Dept. Tech. Physics, Inst. Gen. Chemistry, Warsaw). *Chem. Anal., Warsaw*, 1957, **2** (2), 149–159.—Lithium in calcium hydroxide in concn. of a few μg per g is determined spectrographically after conversion into the sulphate. An a.c. arc of the Swienticki type and carbon electrodes are used. The lines Li 6707·8 A and K 6938·9 A have been chosen. The relative standard error of the mean of 5 to 10 measurements is $\simeq \pm 5\%$. H. DMOWSKA

46. **Determination of excess of zinc in zinc oxide.** H. J. Allsopp (Royal Aircraft Estab., Farnborough, Hants, England). *Analyst*, 1957, **82**, 474–483.—Excess of zinc in zinc oxide is determined by measurement of the hydrogen evolved by treatment of the sample with previously dist. HCl under reduced pressure, complete evolution of gas being ensured by evaporation of the acid soln. to dryness. The apparatus is described and illustrated and the radiochemical standardisation of the method is described. The effect of grinding on the samples is studied. The method is claimed to be sensitive to a 0·1 p.p.m. excess of zinc in the oxide, and reproducibility is good. The excess of zinc in the specimens analysed ranged from 0·2 to 18 p.p.m. A. O. JONES

47. **Improved method for the simultaneous determination of aluminium, copper and magnesium in zinc alloys by spectrographic method.** B. C. Kar, M. K. Gupta and V. Muthukrishnan (Nat. Metallurgical Lab., Jamshedpur). *J. Sci. Ind. Res., B, India*, 1957, **16**, 27–31.—*Procedure*—Soln. (1%) of the alloy in HCl are employed. The upper electrode is a graphite rod, 1·5 in. long and 10 mm in diam., and the lower is a carbon rod 6 mm in diam. with an 80° cone at the tip. A hole 3·4 mm in diam. is drilled along the axis of the graphite electrode to within 1·25 mm of its sparking end, which is bevelled at 45° to give a truncated cone, tip diam. 3·4 mm. The porosity of the graphite electrode is increased by treating it with a d.c. arc at 5 amp. for 15 sec. The cavity is filled with the sample soln. and a spark is passed for 1 min.; the cavity is refilled and sparked for a further 1 min.; then it is filled a third time and exposures are given after 10 sec. of pre-sparking. An accuracy of $\pm 3\%$ is attained with this continuous-feed method. C. A. SLATER

48. **Polarographic analysis of complex calamines. Determinations of iron, lead, zinc, manganese, arsenic and antimony.** S. E. Khalafalla and M. Y. Farah (Chem. Dept., Univ. Cairo). *J. Chim. Phys.*, 1957, **54** (3), 251–257.—A polarographic method of determining Fe, Pb, Zn, Mn and traces of As and Sb in calamine minerals is described. For Fe, Pb and Zn, the supporting electrolyte is HNO_3 - KCl (0·1 *M* each) which was found to give better results than does HCl - KCl or acetic acid - ammonium acetate. This method also enables traces of As and Sb to be detected and determined. For the determination of Mn, the most satisfactory medium is *M* NaCN. Polarograms are reproduced for each determination. S.C.I. ABSTR.

49. **Colorimetric determination of mercury as the double iodide of mercury and copper.** D. P. Shcherbov and K. M. Konovalova (Central Lab. of the Kazakh Geological Dept.). *Zavod. Lab.*, 1957, **23** (6), 663–665.—The method of Finkel'shtein *et al.* (*Zavod. Lab.*, 1954, **20**, 155) has been found unsatisfactory. A possible reason for faulty results is suggested. G. S. SMITH

50. **Application of gas-phase chromatography to the boron hydrides.** J. J. Kaufman, J. E. Todd and W. S. Koski (The Johns Hopkins Univ., Baltimore, Md., U.S.A.). *Anal. Chem.*, 1957, **29** (7), 1032–1035.—Mixtures of B_2H_6, B_4H_{10} and B_5H_9 with small amounts of C_2H_6 are completely resolved in a column of kieselguhr coated with paraffin oil, tricresyl phosphate or "Octoil S" under a flow of He at room temp. Detection is by means of thermal conductivity measurements. Under the conditions of experiment, B_5H_{11} undergoes partial decomposition to B_4H_{10} and B_2H_6. D. A. PANTONY

51. **Characteristics of boron - benzoin complex. Improved fluorimetric determination of boron.** C. E. White and D. E. Hoffman (Univ. of Maryland, College Park, U.S.A.). *Anal. Chem.*, 1957, **29** (7), 1105–1108.—Variables in the fluorimetric determination of B as its benzoin complex are investigated. Of especial interest is its decomposition in the u.v. radiation used for excitation; use of a glycine buffer or of *iso*propylamine, minimum exposure time, and standardisation of the order of mixing of reagents are recommended. The max. excitation is obtained between 310 and 400 mμ to give a max. emission from 450 to 520 mμ. The composition of the complex is established as 1 atom of B per 1 molecule of benzoin. D. A. PANTONY

52. **Spectrophotometric determination of boron in high-temperature alloys by quinalizarin method.** A. H. Jones (General Motors Corp., Detroit, Mich., U.S.A.). *Anal. Chem.*, 1957, **29** (7), 1101–1105.—The sample, containing <0·3 mg of B and <50 mg of Ti, is fused with Na_2O_2 and the cooled melt is

extracted with water. To the soln. is added an excess of H_2SO_4, and the mixture is boiled, then cooled, and to it is added sufficient Na_2SO_3 to reduce Cr^{VI}. After making up the soln. to standard vol., an aliquot is treated with quinalizarin reagent, and after 2 hr. the absorption of the soln. is measured at 615 mμ, with a reagent blank as reference soln. The concn. of B is derived from calibration curves or a nomogram. Reproducibility and accuracy data are presented, and variables associated with the determination are discussed. Any interference due to Ti^{4+} is avoided by use of the calibration curves; F^-, NO_3^- and $Cr_2O_7^{2-}$ do not interfere.

D. A. PANTONY

53. **Chemical analysis of cupola slags. I. Determination of alumina.** W. E. Clarke (B.C.I.R.A., Alvechurch, Birmingham). *J. Res. Brit. Cast Iron Ass.*, 1957, **6** (10), 505–511.—The sample (1 g) is dissolved in a $HClO_4$ - HNO_3 - HF mixture and the Si is volatilised as SiF_4. The soln. is buffered to pH 4·5 with Na tartrate, and Fe and Mn are removed by adding Na diethyldithiocarbamate and extracting with $CHCl_3$. The pH is adjusted to 8·15 with NaOH and 0·1 *N* HCl, excess of KF is added and the liberated KOH is titrated with standard acid.

S.C.I. ABSTR.

54. **The spectroscopic determination of magnesium, zinc, vanadium and chromium content of pure aluminium using a hollow electrode.** L. Erdey, E. Gegus, E. Kocsis (Inst. für Allgemeine Chemie, Tech. Univ., Budapest). *Acta Chim. Acad. Sci. Hung.*, 1957, **11** (3–4), 277–293.—The method previously described (*cf. Anal. Abstr.*, 1956, **3**, 1932) has been applied to the assay of aluminium samples and is compared throughout with the Scheibe - Rivas procedure, previously the best of the solution methods. The line pairs selected were—Mg 2795 - Al 3050, Mg 2790 - Al 3050; Zn 3345 - Al 3050; V 3102 - Al 3050; V 3110 - Al 3050; and Cr 2677 - Al 2652 A. Only Mg could be calculated directly; in all other cases the Kaiser correction for background radiation had to be applied. The method proved to be quicker, simpler and in most cases more accurate and sensitive than that of Schiebe - Rivas. Sensitivity limits were Mg 0·001 to 17·5%, Zn 0·008 to 2%, Cr 0·003 to 2%, and V 0·003 to 1%, with an average error of ±5%. By using an enrichment process, V can be determined at 0·0003% concn.

F. J. M.

55. **Spectrochemical determination of ferric oxide and silicon dioxide in alumina.** D. Conrad (Radio Corp. of America, Harrison, N.J., U.S.A.). *Appl. Spectroscopy*, 1957, **11** (2), 84–86.—The alumina is mixed with spectrographically pure Ge and graphite powder in the ratio 7:15:15 and transferred to a cratered electrode which is burned as the anode in a d.c. arc at 220 V, 10 amp. The addition of Ge yields uniform, reproducible spectral lines. Working curves are prepared with Fe and Si in concn. equivalent to 0·01% to 0·15% of Fe_2O_3 and SiO_2, respectively. The results compare well with those obtained by gravimetric procedures, and the method is less time-consuming.

K. A. PROCTOR

56. **Short cuts in analysing aluminium alloys.** C. Goldberg (New England Smelting Works Inc., West Springfield, Mass., U.S.A.). *Foundry*, 1956, **84**, 118–119.—Time-saving modifications of standard procedures are given. *Procedure for Cu and Si*—Dissolve the sample (0·5 g) in 10 ml of conc. HCl followed later by 10 ml of conc. HNO_3 and 15 ml of conc. $HClO_4$. Heat the mixture to fuming, cool, add 25 ml of H_2O, warm, and collect and wash the SiO_2. To the filtrate add 75 ml of aq. NH_3, 100 ml of ammoniacal ammonium citrate soln. (20% w/v) followed by 20 ml of aq. NH_3 and then H_2O to make 200 ml. Take 100 ml of the soln. and after 5 min. read the extinction at 650 mμ; find the content of Cu from a standard curve. Less than 2% of Fe can be tolerated but allowance must be made for any Ni, Co or Cr. *For Fe and Ni*—To the remaining 100 ml of soln. add 10 ml of HNO_3 (1 + 1) and electrolyse at 5 amp. with stirring. After 5 min. add 50% H_2SO_4 (1 ml), 10% sulphamic acid (1 ml) and 1% HCl (2 drops). When all the Cu has been deposited, dilute the filtrate to 250 ml and determine the Fe photometrically (at 525 mμ) in a 10-ml aliquot by the thiocyanate method and the Ni in a similar aliquot by the dimethylglyoxime method, also at 525 mμ. *For Cr*—Dissolve 1 g of alloy with ≃5 g of NaOH (pellets) in H_2O, adding 30% H_2O_2 dropwise to complete oxidation of the Cr. Filter through a Gooch crucible, washing with 10% aq. NaOH; dilute the clear filtrate to 100 ml with 10% aq. NaOH, mix well and immediately read the extinction of the soln. at 425 mμ. *For Bi*—Dissolve 0·5 g of alloy in 40% aq. NaOH (10 ml); add H_2O (10 ml), 50% H_2SO_4 (30 ml) and 50% HNO_3 (10 ml), and boil until the Cu is dissolved and the excess of HNO_3 is removed. Add 1 g of hydrazine sulphate, boil for 5 min., cool and then add NaF (0·5 g), thiourea (10 to 15 g) and H_2O to make up to 200 ml. Filter through a dry paper and measure the extinction at 425 mμ.

W. J. BAKER

57. **Use of amines in inorganic polarography. V. Simultaneous determination of thallium, copper, lead and cadmium in indium.** J. Doležal (Inst. Anal. Chem., Charles' Univ., Prague, Czechoslovakia). *Chem. Listy*, 1957, **51** (6), 1058–1060.—Thallium (≮0·1%), Cu (≮0·02%), Pb (≮0·07%) and Cd (≮0·03%) in pure indium can be determined polarographically in a soln. of 1 to 2 *M* ethylenediamine and *M* KOH. *Procedure*—Dissolve the sample (250 mg) in $HClO_4$ (30%) (3 to 5 ml) with mild warming. Add two to four drops of conc. HNO_3 and evaporate to 1 or 2 ml. Cool, and adjust the vol. (25 ml) (when necessary, add 1 to 2 ml of $HClO_4$). Measure 2·5 ml into a 10-ml volumetric flask, add 4 *M* ethylenediamine (5 ml) and, when the ppt. is dissolved, add 8 *M* KOH (1·25 ml) and adjust the vol. with H_2O. Remove O and record the polarographic wave from −0·2 V.

J. ZÝKA

58. **Polarographic determination of carbon monoxide.** K. Linhart (Mining Inst., Ostrava-Radvanice C.S.R.). *Chem. Anal., Warsaw*, 1957, **2** (2), 187–192.—Carbon monoxide is oxidised by iodine pentoxide; the optimum temperature is found to be 110°. The liberated iodine is absorbed by KOH or NaOH solution and oxidised by ozone to iodate, which is then determined polarographically. The presence of hydrogen causes interference. The method is applicable to concentrations of CO of the order of 10^{-5} % if enough gas is available.

H. DMOWSKA

59. **Determination of $^{14}CO_2$ in a Geiger - Müller counter.** G. Delibrias. *J. Inorg. Nucl. Chem.*, 1955, **1**, 238–240; *Rap. Cent. Ét. Nucl. Saclay*, 1956, No. 549, 3 pp.—A method for the measurement of small amounts of CO_2 in a Geiger - Müller counter with an addition of ethanol (v.p. of the ethanol ≡ 1 cm of Hg) is described briefly. With

an electronic time constant of the order of 10^{-3} sec., a plateau of several hundred volts is obtained. The min. activity detectable is $\simeq 5 \times 10^{-12}$ curies of ^{14}C in a vol. of CO_2 ranging from 3 to 30 cu. cm at s.t.p. J. M. JACOBS

60. **An ion-exchange method of estimating carbonate in the presence of uranium and vanadium.** A. K. Sundaram, M. Sundaresan and D. G. Vartak (Chem. Div., Atomic Energy Estab., Bombay). *J. Sci. Ind. Res., B, India*, 1957, **16**, 25–27.—*Procedure*—A soln. of Na_2CO_3 ($\simeq 0{\cdot}1\ N$) (25 ml) containing UO_2^{2+} and VO_3^- is passed through a column of Amberlite IRA-400 (CO_3^{2-} form). The carbonate content of the eluate is estimated by titration with standard acid and this amount is equiv. to the original carbonate plus carbonate equiv. to the VO_3^- contained in the soln. In the presence of U (8 mg) and V (10 mg), Na_2CO_3 (20 mg) can be estimated with an accuracy of $\pm 2\%$. C. A. SLATER

61. **Colorimetric determination of large amounts of silica.** R. Petcov and G. Răducanu. *Rev. Chim., Bucharest*, 1957, **8** (2), 259.—The determination of SiO_2 as molybdosilicic acid (**I**) depends on all of **I** being present in the form of the α-acid, *i.e.*, under conditions where $(H^+)/(MoO_4^{2-}) = 1{\cdot}5$ to 2. If the relation is > 3, then the β-acid is formed. *Procedure*—The finely powdered material to be determined (0·2 g) is spread on a solidifying melt of 9 g of KOH and 3 g of KNO_3 in a nickel crucible. The crucible is heated at redness (600°) for 20 min., and, after cooling, the contents are dissolved in boiling water and made up to 1 litre. A 10-ml aliquot is treated with 1·5 ml of 2 *N* HCl and 2 ml of NaF soln. (2·5%). To remove excess of F, 0·3 g of $AlCl_3$ (cryst.) is added after 10 min. and mixed well and, when the $AlCl_3$ is dissolved, 3 ml of ammonium molybdate soln. (5%) is added. The soln. is made up to 100 ml and the colour is measured after 15 min., with a 1-cm cell and S43 filter, or the soln. can be compared with standard picric acid (0·6 g per litre; 1 ml ≡ 1 mg of SiO_2). The advantages claimed for this method are rapidity (90 min.), and the fact that it does not require a platinum crucible. The max. error is $\pm 4{\cdot}6\%$. H. SHER

62. **Determination of low concentrations of lead.** Yu. Yu. Lur'e and Z. V. Nikolaeva ('Vodgeo' Inst.). *Zavod. Lab.*, 1957, **23** (6), 652–655.—Improvements in the dithizone colorimetric titration and the chromate - diphenylcarbazide colorimetric methods are described. With the former method, Cu is separated first by extraction. The sample soln. containing 5 to 10 μg of Pb in a separating funnel is neutralised to phenol red indicator, treated with sufficient *N* HCl to give an acid concn. of 0·01 *N*, and then with 1-ml portions of a 0·2% soln. of dithizone in CCl_4 or $CHCl_3$ with intermediate shaking and removal of the organic solvent layer until this remains green. The Cu-free soln. is treated with Na_2CO_3 soln. until the colour is orange (pH 6·8 to 7·4), 1 ml of 10% $K_4Fe(CN)_6$ soln., 2 ml of 1% hydroxylamine hydrochloride, 2 ml of 10% Pb-free ammonium citrate, citric acid or Na tartrate and, after mixing, Na_2CO_3 soln. to give a crimson colour (pH 8·5 to 9). The soln. is then extracted with 1-ml portions of a standardised soln. ($\simeq 0{\cdot}2$ %) of dithizone, the solvent layer being collected in a different test-tube each time until the extract is green or an intermediate colour. The amount of titrant can thus be found to within $\simeq 0{\cdot}5$ ml. A blank experiment is made at the same time and the amount required is allowed for. The dithizone soln. is standardised under similar conditions against a soln. containing 5 μg of Pb per ml. Interference of Zn is prevented by the use of the stated amount of $K_4Fe(CN)_6$. With the second method, the soln. containing < 10 μg of Pb is neutralised and treated with 1 ml of dil. H_2SO_4 (1 + 99) for every 100 ml of soln., followed by 0·5 ml of a suspension of ZnS (prepared by mixing a soln. of 25 g of $ZnCl_2$ and a soln. of 42 g of $Na_2S.9H_2O$, washing the ppt. with water and then shaking it with 200 ml of water). After 1 hr. of intermittent stirring the ppt. is collected, washed and returned to the beaker, where it is dissolved in 3 ml of dil. HCl (1 + 3). After being boiled to remove H_2S, the soln. is treated with 2 to 3 drops of conc. HNO_3, evaporated to a vol. of 1·5 to 2 ml and transferred to a 10-ml centrifuge tube for pptn. of $PbCrO_4$ by means of 1 ml of 30% K_2CrO_4 added to the soln. made slightly acid with acetic acid, or of $K_2Pb(CrO_4)_2$ (preferred for greater sensitivity and accuracy but requires greater care in the regulation of pH) by adding to the soln. ($\gg 4$ ml) 150 to 200 mg of NH_4Cl, then aq. NH_3 to give a red colour with phenol red indicator, followed by dil. acetic acid (1 + 1) to give an orange colour (pH 6·8 to 7·4), 1 ml of 40% ammonium acetate, and 1 ml of 30% K_2CrO_4. In either case the soln. is centrifuged after several hours, preferably on the following day, and the ppt. is washed by centrifuging with 0·4% ammonium acetate soln. (3 × 4 ml). The ppt. is dissolved in 3 ml of dil. HCl (1 + 3) and the soln. in a flat-bottomed tube is diluted to 5 ml and treated with 10 ml of 0·02% aq. diphenylcarbazide soln. The colour intensity is measured after 10 min. G. S. SMITH

63. **Determination of lead in lead fluoroborate solutions.** G. Gabrielson (AB Tudor, Nol, Sweden). *Metal Finish.*, 1957, **55** (3), 56–57.—To the sample soln. (≡ ≃55 g of Pb per litre) is added a known excess of 0·1 *M* EDTA (disodium salt). The soln. is buffered to pH 10·0 and the excess of reagent is determined by titration with 0·1 *M* $ZnCl_2$, with Eriochrome black T as indicator. There is a sharp colour change, from blue to red, at the end-point. S.C.I. ABSTR.

64. **Spectrochemical analysis of corrosion products on lead sheath of cables.** W. J. Bennett (Northern Electric Co. Ltd., Lachine, Quebec, Canada). *Appl. Spectroscopy*, 1957, **11** (2), 73–75.—A procedure for the spectrographic determination of a variety of elements in a lead matrix over a concn. range of 0·01% to 10% and of Pb as the main component in the 30% to 90% range is described. The powdered sample is buffered with GeO_2 and graphite powder and the resulting mixture pelleted. Two spectra of widely different sensitivities are recorded for each sample pellet under widely different discharge conditions. Duplicate analyses can be made with a 15-mg sample. K. A. PROCTOR

65. **Detection of titanium with sulphosalicylic acid and tetraphenylarsonium salt.** M. Ziegler and O. Glemser (Inst. für Anorg. Chem., Göttingen Univ., Germany). *Z. anal. Chem.*, 1957, **157** (1), 17–18.—To detect Ti^{4+}, a few drops of aq. sulphosalicylic acid (10%) and solid tetraphenylarsonium chloride (15 to 30 mg) are added to the sample soln. Sufficient acetate buffer (pH 4·6) is added to dissolve the white ppt., and the mixture is shaken with a small vol. of $CHCl_3$. A clear yellow coloration (in

daylight) of the $CHCl_3$ extract indicates the presence of Ti^{4+}. The lower limit of sensitivity is 0·05 μg of Ti in a limiting concn. of 1 in 20×10^6; most common metal ions (Ni^{2+}, Co^{2+}, Mn^{2+}, Zn^{2+}, Al^{3+}, Mg^{2+}, Ca^{2+} and Fe^{2+}) do not interfere. Tetraphenylphosphonium chloride can also be used.

W. J. BAKER

66. **Quantitative determination of zirconium by ethanolamine.** I. P. Ryazanov and V. P. Milin. *Uch. Zap. Saratov. Univ.*, 1956, **43**, 155–158; *Ref. Zhur., Khim.*, 1957, Abstr. No. 27,167.—For the pptn. of $Zr(OH)_4$, ethanolamine (**I**) is suggested. Take $\geq$0·04 g of Zr^{4+} salt in 40 to 50 ml of soln., heat to between 85° and 90°, acidify with 25% HNO_3 and precipitate the $Zr(OH)_4$ with 10 ml of 5% **I** soln. After 10 to 15 min. filter off the ppt., wash with 3 to 5 portions of hot 1% **I** soln. and ignite at 800° to 900° to ZrO_2. The error is $<$0·25%.

L. HERDAN

67. **Use of benzenesulphinic acid for determining zirconium.** I. P. Alimarin and S. S. Alikberov (M. V. Lomonosov Moscow Inst. of Fine Chem. Technol.). *Zavod. Lab.*, 1957, **23** (6), 658–660.—The quant. pptn. of Zr as $ZrO(C_6H_5SO_2)_2$ occurs with Na benzenesulphinate in the presence of $\geq$1·5 *N* HCl (or HNO_3) if the reagent concn. is 2%. The solubility of the ppt. at 20° in water is $1{\cdot}3 \times 10^{-6}$ *M*. There is no interference from Al, Be, Ni, Cr, Nb, Ta and small amounts of Fe. Rare earths do not interfere unless their concn. exceeds ten times that of the Zr; a re-pptn. is then desirable. Pptn. is carried out in 0·5 *N* HCl and if a re-pptn. is required the ppt. is dissolved in 4·2 ml of conc. HCl and the soln. is again treated with the reagent. The washed ppt. is ignited to ZrO_2 and weighed. A single pptn. of 6 to 25 mg of ZrO_2 in the presence of 10 mg of Fe gives a ppt. containing $<$0·01 mg of Fe. Interference of Fe, *e.g.*, in steel, can be prevented by prior extraction of the $FeCl_3$ by an organic solvent or by reduction of Fe^{3+} to Fe^{2+} with SO_2 or ascorbic acid.

G. S. SMITH

68. **Complexometric titration of zirconium and hafnium.** N. S. Poluéktov, L. I. Kononenko and T. A. Surichan. *Zavod. Lab.*, 1957, **23** (6), 660–662.—The soln. (10 ml) containing a salt of Zr (0·5 to 40 mg) is mixed with HCl to give an acid concn. of between 1·5 and 2·2 *N* and boiled, and then treated with 0·2 ml of a 0·02% soln. of *p*-nitrophenylazocatechol as indicator. The indicator gives a crimson-red colour with Zr. The soln. is titrated from a micro-burette with 0·05 *M* EDTA (disodium salt) until the colour of the soln. becomes yellow, whereupon the soln. is again heated to boiling and treated with one or two drops of indicator, and the titration is continued if a red colour has developed. EDTA combines with Zr in the ratio 1:1. No interference is caused by Fe^{2+}, Th, Ti, Sn^{4+}, Mo, Nb, Al, Ca, Bi, Sb, Cu, Ni, Ge, rare earths, etc., in amount similar to that of the Zr. Interference is caused by Fe^{3+}, V, Ta, W, strong oxidising and reducing agents, tartaric acid $>$10 mg, H_2SO_4 $>$20 mg, oxalic acid, PO_4^{3-} and F^-. Hafnium behaves like Zr. The titre of the EDTA soln. must be established against a compound of Zr containing a similar amount of Hf as the sample to be analysed.

G. S. SMITH

69. **Hydrazine sulphate as a volumetric reagent (hydrazinometry). VI. New volumetric determination of nitrites suitable for the control of drugs.** I. Marxová and J. Zýka (Inst. Anal. Chemistry, Charles' Univ., Prague, Czechoslovakia). *Českosl. Farm.*, 1956, **5** (4), 218–221.—The assumed course of the reaction of hydrazine and its salts with nitrites in acidic soln. is—

$$N_2H_4 + 2HNO_2 \rightarrow N_3 + H_2N_2O_2 + 2H_2O$$
$$H_2N_2O_2 \rightarrow N_2O + H_2O,$$

and a direct potentiometric titration of hydrazine in 10% HCl with the sample containing nitrites is based on this reaction. The reverse titration gives erroneous results, as nitrite is decomposed in the acid medium more quickly than it reacts with hydrazine. There is a considerable change in potential (about 300 mV) at the equivalence point. The soln. of hydrazine sulphate used as standard is very stable.

J. VOLKE

70. **Arsenometric determination of nitrite and ammonium ions.** A. Kellner, C. Szabó and L. Szekeres (Tech. Univ., Budapest, Hungary). *Z. anal. Chem.*, 1957, **157** (1), 13–17.—The proposed two procedures for the determination of milligram amounts of NO_2^- and NH_4^+ depend on oxidation with Br^- and BrO^-, respectively. An excess of $KBrO_3$ - KBr soln. (0·05 to 0·1 *N*) is added to the sample soln., which should be adjusted to between 0·05 and 0·1 *N* HCl. After 15 to 30 min. (and making the soln. alkaline for the hypobromite oxidation) the excess of Br^- or BrO^- is back-titrated with 0·05 to 0·1 *N* As_2O_3 soln., with 0·1 *N* KI or $KH(IO_3)_2$, plus starch soln., as indicator.

W. J. BAKER

71. **Analysis of phosphating solutions: colorimetric determination of nitrate.** L. O. Gilbert (Rock Island Arsenal, Ill., U.S.A.). *Metal Finish.*, 1957, **55** (4), 66–67.—A rapid and accurate method of determining NO_3^- by a modified brown-ring procedure is described. The colour developed (on mixing the reagents completely) is measured by transmittance at 520 mμ in a spectrophotometer. A calibration curve prepared from a series of standards is given.

S.C.I. ABSTR.

72. **The volumetric determination of phosphates and of metals in the presence of phosphates. II. The complexometric determination of several metals in the presence of phosphate with dithizone as indicator.** G. M. Lukaszewski, J. P. Redfern and J. E. Salmon (Battersea Polytechnic, London). *Lab. Practice*, 1957, **6** (7), 389–391.—The method of Wänninen and Ringbom (*cf. Anal. Abstr.*, 1955, **2**, 2360) can be slightly modified for the determination of Zn, Ga, In, Cd, Ni, Co or Al, or $\geq$0·1 millimole of Bi, both in the presence and absence of PO_4^{3-}, or for Th only in the absence of PO_4^{3-}. *Procedure*—To the sample soln. containing $\simeq$0·1 millimole of the metal being determined add a two to three-fold excess of 0·01 *M* EDTA (disodium salt) and warm the mixture at $\simeq$90° for 10 min. Adjust the pH to between 4 and 5 (dil. HNO_3 or aq. NH_3) and then to pH 4·5 by adding 10 ml of buffer soln. (*M* acetic acid - *M* ammonium acetate) and 10 ml of ethanol. Add 2 ml of a freshly prepared 0·025% (w/v) ethanolic soln. of dithizone, and titrate with a 0·01 *M* soln. of $ZnSO_4$ until the colour changes sharply from greenish yellow to red. The accuracy is high.

W. J. BAKER

73. **Determination of pyrophosphate in commercial tripolyphosphate.** H. J. Weiser, jun. (Procter & Gamble Co., Cincinnati, Ohio, U.S.A.). *J. Amer. Oil Chem. Soc.*, 1957, **34** (3), 124–127.—The

method is based on anion exchange, elution by four different eluents giving quantitative separation, acid hydrolysis of eluate aliquots with H_2SO_4 to orthophosphate and estimation of P_2O_5 photometrically with molybdate solution. The eluents used are—0·05 *M* HCl - 0·1 *M* KCl; 0·2 *M* KCl; 0·4 *M* KCl; and 0·75 *M* KCl, all except the first being buffered to pH 5. When applied successively to the column, they elute, respectively, ortho-, pyro-, tri- and trimeta-phosphates. G. HELMS

74. **Analysis of mixtures of the condensed phosphates by ion-exchange chromatography.** T. V. Peters, jun. (Rutgers Univ., New Brunswick, N.J., U.S.A.). *Dissert. Abstr.*, 1956, **16** (10), 1809.—Methods are developed for analysis of mixtures containing ortho-, pyro-, tri-, trimeta-, tetra- (**I**), and tetrameta-phosphate (**II**), and Graham's salt (**III**). Except for **I** and **II**, the lower phosphates are eluted in separate eluate fractions from columns of strong base anion-exchange resins with KCl eluents buffered at pH 5, total PO_4^{3-} being determined spectrophotometrically in each fraction. **I** and **II** are obtained in the same eluate fraction at this pH and can be determined by potentiometric titration after removal of buffer. **III** is depolymerised and eluted from the column, after removal of the lower phosphates, with 3 *M* HCl.
S.C.I. ABSTR.

75. **Determination of tripoly- and pyro-phosphate in tripolyphosphate.** R. Raemaekers (Usines Kuhlmann de Rieme-Selzaete, Belgium). *Chim. Anal.*, 1957, **39** (7), 254–259.—If Raistrick's method (*cf. Brit. Abstr. C*, 1951, 328) is used for the analysis of commercial $Na_5P_3O_{10}$, the compositions of the co-pptd. $NaZn_2P_3O_{10}.9H_2O$ and $Zn_2P_2O_7.H_2O$ deviate from the theoretical, although the relation between the contents of $P_3O_{10}^{5-}$ and $P_2O_7^{4-}$ and the content of ZnO in the ppt. is linear. In the modified method the commercial sample and pure $Na_5P_3O_{10}.6H_2O$ are analysed concurrently by pptn. of the sodium zinc salts. The origin of the working curve is thus obtained, its "slope factor" (1% increase in ZnO content = 8% $P_2O_7^{4-}$) being known. The error is within 1%; neither PO_4^{3-} (>2%) nor PO_3^{3-} (>3%) interfere, but any hexametaphosphate is weighed as $Na_5P_3O_{10}$. The sodium zinc salts are pptd. with Zn acetate (in HCl soln.) and 0·1 *N* NaOH at ≃20° and pH 3·8, followed by the addition of acetone (30 ml) to ensure min. solubility of the ppt. The ppt. is collected, dried and ignited (30 min. at 800°) and, after weighing, it is dissolved in hot 4 *N* HCl. The content of ZnO is then determined by titration of the buffered soln. with EDTA (disodium salt) and Eriochrome black T indicator.
W. J. BAKER

76. **Polarographic determination of arsenic.** King-Hung Tsao, Yun-Chieng Loo and Teng-Han Tang. *Acta Pharm. Sinica*, 1957, **5** (1), 9–16.—The polarographic behaviour of As^{3+} in HCl soln. was studied. In 1 to 3 *M* HCl, As^{3+} showed two well-defined waves. The first wave was the more suitable for quant. analysis, the diffusion current being proportional to the concn. of As^{3+} within the range 10^{-6} to 2×10^{-3} *M*. Addition of traces of gelatin disturbed the wave form and caused the half-wave potential to shift to a more negative value. With gelatin concn. greater than 0·02%, the two polarographic waves merged into a spread-out wave, rendering the half-wave potential and diffusion current immeasurable. S. H. YUEN

77. **Colorimetric method for determination of antimony (iodide method) and tin (molybdenum blue method).** Z. Marczenko (Inst. Gen. Chem., Warsaw). *Chem. Anal., Warsaw*, 1957, **2** (2), 160–167.—A method is described for the determination of small amounts of Sb and Sn in the presence of a large excess of Cu. The Cu is separated from Sn and Sb as the sulphide in alkaline solution. The filtrate is acidified and made up to volume. Antimony is estimated as $HSbI_4$ by measuring the colour which is developed on the addition of 50% KI to an aliquot of filtrate. The colour, which remains stable for 1 hr., is measured by a photo-electric absorptiometer, with 2-cm cells and blue filters, and the concn. of Sb is read from a standard graph. The presence of Bi causes high results, but interference due to Sn is negligible, and As does not interfere. Tin is estimated by measuring the colour of the blue complex formed by the addition of sodium silicate - ammonium molybdate reagent to an aliquot of the filtrate, which contains Sn previously reduced to the stannous state by boiling with HCl and granular zinc. The colour, which remains stable for 3 hr., is measured by the photo-electric absorptiometer with 2-cm cells and red filters, and the concn. of Sn is calculated from a standard graph. Antimony and As, if of the same order, do not interfere. H. DMOWSKA

78. **Absorptiometric determination of traces of iron in bismuth.** D. G. Holmes (Chem. Engng Div., A.E.R.E., Harwell, England). *Analyst*, 1957, **82**, 528–529.—The sample (up to 2 g) is dissolved in HNO_3 and the soln. is evaporated to dryness three times with HCl. The residue is dissolved in the least amount of HCl, and 1 ml of hydroxylamine hydrochloride soln. (5% w/v) is added. The liquid is then treated with a mixture of EDTA (disodium salt), Na citrate and *o*-phenanthroline soln. and adjusted to a definite vol. After 1 hr. the extinction is measured in a Spekker absorptiometer with an Ilford No. 603 filter. The absorptiometer is calibrated with a mixture of standard soln. of Bi and Fe treated similarly. Iron may occur in bismuth to the extent of 5 to 50 p.p.m.
A. O. JONES

79. **The analysis of bismuth - uranium alloys.** R. A. J. Shelton (Metallurgy Dept., Manchester Univ., England). *Analyst*, 1957, **82**, 531–532.—The soln. of alloy in dil. HNO_3 is evaporated to dryness, the residue is evaporated with conc. HCl and then dissolved in conc. HCl, diluted and treated with excess of EDTA. The pH is adjusted to 5·5 ± 0·3 by addition, after neutralisation, of an acetic acid - ammonium acetate mixture and the U is pptd. at ≃60° by addition of 4% ethanolic oxine soln. The collected ppt. is washed with a warm 40-fold aq. dilution of the oxine soln., dried at 105° and weighed. The U-free filtrate is treated with more oxine soln., warmed to 50° and made alkaline with NaOH soln. The pptd. Bi oxinate is collected, washed, dissolved in hot dil. HCl and re-pptd. by addition of oxine soln. and a slight excess of aq. NH_3, then collected, washed, dried and weighed. The method is particularly applicable to alloys in which the Bi to U ratio lies between 3:1 and 1:3.
A. O. JONES

80. **Polarographic determination of oxygen.** K. Linhart and J. Žagmen (Mining Inst., Ostrava-Radvanice, ČSR). *Chem. Anal., Warsaw*, 1957, **2** (2), 183–186.—The basis of the determination is the oxidation of the chromous ion. A measured amount

of the gas under examination is introduced into $CrCl_2$ soln. which has been standardised polarographically. After the reaction the residual Cr^{3+} are again determined polarographically. The method is standardised with a known amount of oxygen. From the difference in the wave-heights, the amount of oxygen is calculated.

H. DMOWSKA

81. **Determination of carbon in purified sulphur.** F. Fehér and K. H. Sauer (Chem. Inst. Univ. Köln, Germany). *Z. Naturforsch.*, 1957, **12b** (1), 65.—The sulphur is completely burnt in a stream of O and the CO_2 generated in the process from contaminating carbon is eliminated from the gas by passing it through dilute alkali. The CO_2 content of the alkaline solution is measured by its conductivity. To obtain reproducible and accurate results it is necessary (*a*) to admit the gas to the alkali at a regular rate, *e.g.*, by a pump which forces the gas up through the solution; and (*b*) by arranging the electrodes in such a manner that one is always surrounded by fresh alkali whereas the other remains in contact with the gas-containing alkali. An automatic recorder registers the carbon content directly. Sulphur oxides must be removed from the gas completely before its admission to the alkali. This is achieved by mixing the sulphur with a tenfold quantity of lead oxide before combustion and by passing the cooled gas (−50°) through a H_2SO_4 - H_2O_2 trap followed by an exchange column. No sulphur was detected in the alkali after 20 determinations. E. KAWERAU

82. **Rapid absorptiometric determination of sulphide in sulphate black liquor.** J. D. H. Strickland and J. B. Risk. *TAPPI*, 1957, **40**, 91–94.—The determination of sulphide by the Lauth violet absorptiometric method has been adopted for routine measurement of Na_2S in weak black liquor before and after air oxidation. The method is speedy and simple, and is particularly suitable for very low sulphide concn. Results agree well with those of the Borlew and Pascoe method.

S. V. SERGEANT

83. **Quantitative analysis of spent sulphuric acid from alkylation processes.** V. D. Yasnopol'skiĭ, I. É. Dolnakova and N. I. Pervova (Azerbaijan Sci. Res. Inst. of the Petroleum Refining Industry). *Zavod. Lab.*, 1957, **23** (6), 665–667.—Free H_2SO_4 in the presence of sulphonic acids is determined by adding 0·5 g of the sample to 50 ml of ice-cold water, addition of a small excess of $BaCO_3$ and weighing the $BaSO_4$ pptd. Sulphonic acids are determined by neutralising the sample with $BaCO_3$, dilution, and filtering off the $BaSO_4$, evaporating the filtrate to dryness and drying to const. wt., igniting the residue to $BaSO_4$, and weighing.

G. S. SMITH

84. **The determination of colouring matter in glass by surface treatment. (Determination of chromium.)** N. A. Tananaev and L. I. Ganago. *Trudy Ural'sk. Politekhn. Inst.*, 1956, (57), 73–75; *Ref. Zhur., Khim.*, 1957, Abstr. No. 30,995.—Place two drops of HF soln. in a wax-ringed area of the surface of the cleaned sample and standard glasses. After 5 min. add 3 or 4 drops of water, transfer the soln. to a platinum crucible, and boil with 3 or 4 drops of H_2SO_4 (1:1) to obtain a clear soln. Add 2 drops each of a 0·1% soln. of $AgNO_3$, a 0·05% soln. of $MnSO_4$ and a saturated soln. of $(NH_4)_2S_2O_8$, boil till no more O is evolved, transfer the soln. to a cylinder with 4 or 5 drops of H_2SO_4 (1:3) and 8 to 15 drops of a 0·2% soln. of diphenylcarbazide in ethanolic acetic acid. Compare the colours formed.

C. D. KOPKIN

85. **Photometric determination of chromic oxide by means of EDTA (disodium salt) in chrome magnesites and in chromium ores.** A. M. Pinus (Satkinskiĭ Works 'Magnezit'). *Zavod. Lab.*, 1957, **23** (6), 662–663.—The method is suitable for determining large amounts of Cr in the presence of much Fe. The sample (0·05 g) is fused with $KHSO_4$, the cooled melt is dissolved in hot water with the addition of 10 ml of conc. HCl, the cooled soln. is diluted to 100 ml in a calibrated flask, and a 20-ml aliquot is treated with 2 ml of a 20% soln. of Na K tartrate, 3 ml of 5% EDTA (disodium salt) soln., and 4 drops of phenolphthalein indicator. After the addition of aq. NH_3 (1 + 1) to give a pale-pink colour and 5 ml of 2 *N* acetic acid, giving a pH of 4, the soln. is boiled for 5 min., cooled and diluted to 50 ml in a calibrated flask, and then examined on an absorptiometer with a green filter. G. S. SMITH

86. **Colorimetric determination of molybdenum in scheelite ores and concentrates.** R. P. Hope (King Island Scheelite Ltd., King Island, Australia). *Anal. Chem.*, 1957, **29** (7), 1053–1055.—The material (0·2 to 2 g) is fused with NaOH and the cooled melt is extracted in hot water. After filtration, the soln. is made up to standard vol. and an aliquot containing 0·05 to 0·5 mg of MoO_3 is treated with conc. ammonium citrate followed by dil. HCl, conc. aq. NH_4SCN and KI (as reducing agent) and then Na_2SO_3. After 30 min. the absorption is measured at 460 mμ, and the concn. of Mo is derived from standards or a linear calibration curve. A standard deviation of 0·03 on a mean molybdenum content of 2·63% (15 determinations) is given. The effect of variables is discussed, and it is stated that up to 50 mg of W^{VI}, or 5 mg of Sb^{3+} or Sb^{5+} can be tolerated. D. A. PANTONY

87. **Colorimetric determination of microgram amounts of tungsten in uranium - tantalum - tungsten alloys.** C. E. Bricker and G. R. Waterbury (Univ. of California, Los Alamos Sci. Lab., N.M., U.S.A.). *Anal. Chem.*, 1957, **29** (7), 1093–1095.—The alloy is treated with water, H_2SO_4 and HNO_3, followed by HF until solution is complete, and the mixture is diluted to known vol. An aliquot, containing 10 to 250 μg of W and an amount of U approx. the same as that used for a calibration curve, is evaporated to fumes with H_2SO_4. This soln. is made up to 10 ml with a soln. of quinol (in conc. H_2SO_4) and conc. H_2SO_4. The absorption is measured at 478 mμ *vs.* a reagent blank. The tungsten concn. is derived from calibration curves. The coeff. of variation is ±1·1% at the 0·05 to 0·15-mg level. The effects of 31 ions on the determination are listed. D. A. PANTONY.

88. **Ultra-violet spectrophotometric determination of uranium.** B. E. Paige, M. D. Elliott and J. E. Rein (Phillips Petroleum Co., Idaho Falls, U.S.A.). *Anal. Chem.*, 1957, **29** (7), 1029–1032.—The soln. containing 0·01 to 0·1 mg of U per ml is treated with $NaNO_3$ as salting agent and adjusted to pH 3 with HNO_3 or NaOH. The mixture is extracted with tributyl phosphate (25% in *iso*octane) and the absorption of the extract is measured against a reagent blank at 250 mμ; the concn. of U is derived from calibration curves. Modifications are suggested for concn. of >0·1 and <0·01 mg per ml.

The effects of variables are discussed and the coeff. of variation is given as $<1\%$. Ions that form complexes with uranium, Ce^{3+}, Fe^{3+}, Mn^{2+}, Hg^{2+}, Sn^{2+}, Ti^{3+}, Zr^{4+}, MoO_4^{2-}, MnO_4^-, WO_4^{2-} and VO_3^- cause interference unless preliminary separations are carried out. D. A. PANTONY

89. **Electro-deposition of uranium at the microgram level.** C. L. Rulfs, A. K. De and P. J. Elving (Dept. of Chemistry and Engng Res. Inst., Univ. of Michigan, Ann Arbor, U.S.A.). *J. Electrochem. Soc.*, 1957, **104** (2), 80–83.—Recovery of microgram quantities of U by electro-deposition was studied by using ^{233}U as a tracer. Optimum results were obtained in an ammonium oxalate medium, with electrolysis at 80° to 85°, carried out first in acid, and then in alkaline, soln. S.C.I. ABSTR.

90. **Absorptiometric determination of uranium in solutions by an improved thiocyanate method.** H. T. Tucker (Hartebeestfontein Gold Mining Co. Ltd., Stilfontein, Transvaal). *Analyst*, 1957, **82**, 529–530.—In the method for the determination of U by means of the intense yellow colour produced with NH_4SCN, interference by Fe^{III} can be prevented by the addition of $SnCl_2$ if the Fe present does not exceed 5 mg. In the improved method, ascorbic acid is used as the reducing agent and larger amounts of Fe^{III} can then be tolerated, and the acidity of the soln. is not critical within the pH range of 0·5 to 2·5. An aliquot of the sample soln. containing $\simeq 1$ mg of U is heated with 5 ml or more of 2% aq. ascorbic acid soln. and 7 ml of 50% NH_4SCN soln. The extinction is measured within 1 hr. at 365 mμ against a reagent blank, and referred to a calibration graph prepared by similar treatment of standard soln. The method cannot be applied in the presence of Mo, V, Ti, Cr, Os or Ru. A. O. JONES

91. **Estimation of free nitric acid in uranyl nitrate by ion exchange.** D. V. Bhatnagar (Chem. Div., Atomic Energy Estab., Bombay). *J. Sci. Ind. Res., B, India*, 1957, **16**, 23–25.—*Procedure*—A soln. of $UO_2(NO_3)_2.6H_2O$ (0·2%) (25 ml) is passed through a column of Amberlite IR-120 (H^+ form) at a rate of 3 ml per min., and the column is then washed with water (75 ml). The eluate is titrated against standard alkali and the free acid value is obtained by subtracting the amount of acid equiv. to UO_2^{2+} from the titration value. When the ratio of $UO_2(NO_3)_2$ to free acid is 50:1, the accuracy is $\pm 0{\cdot}5\%$. C. A. SLATER

92. **Detection of plutonium (^{239}Pu) by irradiation in the pile.** H. A. C. McKay (A.E.R.E., Harwell, England). A.E.R.E. Report, C/M 23, 1957, 4 pp.—By using a neutron flux of 10^{13} per cm^2 per sec. the direct observation of fissions would give an increase in sensitivity of 10^4 over direct α-counting. A factor of about 50 could be achieved if the radio-tellurium, radio-iodine or radio-barium were determined. Both methods depend on the ratio of U to Pu activities being less than 0·001. G. J. HUNTER

93. **The thermogravimetry of some plutonium compounds.** J. K. Dawson and R. M. Elliott (A.E.R.E., Harwell, England). A.E.R.E. Report, C/R 1207, 1957, 21 pp.—Thermogravimetric curves are given to illustrate the following reactions—PuF_3 and PuF_4 are converted into PuO_2 by heating to between 650° and 700° in moist air; $Pu(SO_4)_2$ is stable between 450° and 650°, but above this range it is converted into PuO_2; $Pu(IO_3)_4$ is rapidly converted into PuO_2 above 500°. It is necessary to heat $Pu(C_2O_4)_2.6H_2O$ above 600° to achieve complete conversion into PuO_2. The conversion of $Pu_2(C_2O_4)_3.10H_2O$ into PuO_2 was achieved at 700° in air and at 850° in vacuum. G. J. HUNTER

94. **Determination of neptunium-239 counting efficiency.** W. S. Lyon (Oak Ridge Nat. Lab., Tenn., U.S.A.). *Anal. Chem.*, 1957, **29** (7), 1048–1050.—Samples of U_3O_8 (uranium enriched to 99·999% ^{235}U) are subjected to various times of neutron irradiation and then examined in a 4π β-methane flow-type proportional counter in which the 0·076 meV γ-rays from the ^{239}U are also counted with a thallium-activated NaI γ-ray spectrometer. The absolute disintegration rate of ^{239}U is determined from coincidence counting of the β- and γ-radiation by use of a given formula, and allowance is made for this in the subsequent β-count of the ^{239}Np activity. The β-counter was shown to have an efficiency of 100%. D. A. PANTONY

95. **Amperometric titration of fluorine as thorium fluoride.** G. D'Amore and G. Faraone (Inst. of Physical Chem. and General Chem., Messina Univ.). *Ann. Chim., Roma*, 1957, **47** (2), 142–149.—The most suitable conditions for amperometric titrations of F^- with $Th(NO_3)_4$ soln. have been determined polarographically and are—(*i*) in 10^{-1} *M* KNO_3, −1·30 V, 0·02% gelatin; (*ii*) in 10^{-1} *M* KCl, −1·40 V, 0·03% gelatin. A constant value for the diffusion current between −1·20 V and −1·40 V is obtained only in the presence of gelatin. The equivalence point corresponds to 1 Th^{4+} per 4 F^-. Concn. of KNO_3 or KCl soln. can vary from 1 to 10^{-3} *M* without affecting the accuracy of titration. High concn. lower the diffusion current value but permit more accurate titration; the most convenient concn. is 10^{-1} to 10^{-2} *M*. The presence of air in soln. does not give rise to errors but electrolytic H should be bubbled through the soln. Variation of temp. (25° ± 0·1°) by ±1° does not influence accuracy; the pH can vary from 1 to 7. Interference by 18 extraneous ions is studied and results are tabulated. L. KRUSZYNSKA

96. **Chromatographic semi-micro analysis of gases. XIII. Separation of chlorine, bromine and iodine.** J. Janák, M. Nederost and V. Bubeníková (Inst. Petrol. Res., Brno). *Chem. Listy*, 1957, **51** (5), 890–894.—The characteristic elution vol. (chromatographic spectra) of chlorine, bromine and iodine on silica gel at temp. from 15° to 140° have been measured. The elution was carried out with N or CO_2 and the halogens were determined polarographically in the eluates from the increase of the Ti^{IV} wave after absorption in a Ti^{III} soln. Bromine can be determined in the presence of a large excess of Cl. J. ZÝKA

97. **Control of the purity of iodine and iodides.** A. Berka and J. Zýka (Inst. Anal. Chem., Charles' Univ., Prague, Czechoslovakia). *Českosl. Farm.*, 1957, **6** (2), 110–113.—N-Bromosuccinimide is applied as an oxidising agent to the titration of iodine, iodides, hydrazine, hydrazides and arsenites. In the determination of iodine, ICl does not interfere, nor do Br^- and Cl^- in the determination of I^-. The titration (which may be performed on the micro scale) is carried out in acid soln. (HCl), with potentiometric or visual (methyl red) indication. J. VOLKE

98. **Reduction of aqueous iodine by trace impurities.** J. H. Wolfenden (Dartmouth College, Hanover, N.H., U.S.A.). *Anal. Chem.*, 1957, **29** (7), 1098–1100.—The use of the absorption max. of the tri-iodide ion in aq. soln. at 353 mμ for the determination of iodide in the presence of iodine, and *vice versa*, is discussed, special reference being made to the influence of external factors. Reduction, especially by atmospheric dust, is held responsible for low results obtained in colorimetric assays at this maximum. D. A. PANTONY

99. **Method for the determination of ferrous iron in rocks and minerals.** A. D. Wilson (Geol. Surv. & Museum, S. Kensington, London). *Bull. Geol. Surv. G.B.*, 1955, **9**, 56–58.—Place the powdered sample (0·5 g) together with solid ammonium metavanadate (usually 0·1 g) in an 80-ml polyethylene or platinum vessel and add 40% HF (10 ml). Allow to stand until the attack is complete (usually 24 hr.), then add 10 *N* H_2SO_4 (30 ml), wash with saturated boric acid soln. (250 ml) into a beaker containing 5 drops of Ba diphenylaminesulphonate indicator, stir until any white curdy ppt. dissolves, and titrate with *N*/30 ferrous ammonium sulphate. R. A. HOWIE

100. **The separation of iron from aluminium by extraction as tributylammonium hexathiocyanatoferrate(III).** M. Ziegler and O. Glemser (Inst. für Anorg. Chem., Göttingen Univ., Germany). *Z. anal. Chem.*, 1957, **157** (1), 19–20.—The extraction procedure used in the photometric determination of Fe with tributylammonium hexathiocyanatoferrate (*cf. Ibid.*, 1957, **154**, 81) can be used for the separation of 1 to 30 μg of Fe from Al. The Al can then be determined gravimetrically. The procedure is applicable to separations in the course of the usual pptn. methods or in place of or for improving the extraction of $FeCl_3$ with diethyl ether.
W. J. BAKER

101. **Separation of iron from aluminium and magnesium by extraction as iron nitrosonaphthoxide.** A. K. Babko and G. T. Mikhal'chishin. *Ukr. Khim. Zhur.*, 1956, **22** (5), 676–687; *Ref. Zhur., Khim.*, 1957, Abstr. No. 31,000.—A method is evolved for the separation of Fe from Al and Mg by the $CHCl_3$ extraction of the complex of Fe^{3+} with 1-nitroso-2-naphthol (**I**). Small quantities of Mg and Al may be determined in the soln. after the removal of Fe. To the soln. to be analysed, treated with aq. NH_3 or HNO_3 to pH ≃1·5, add carefully and with stirring a freshly prepared acetone soln. of **I**, containing 1·3 times the quantity of **I** required for the pptn. of all the Fe. Shake the mixture periodically for 10 min., add $CHCl_3$ and shake for 1 or 2 min. After two extractions, add a few ml of the soln. of **I** and repeat the extraction (× 3). C. D. KOPKIN

102. **Spectrophotometric determination of iron chelates. II.** D. G. Hill-Cottingham (Res. Sta., Long Ashton, Bristol, England). *Analyst*, 1957, **82**, 524–525.—The spectrophotometric method previously described (*Analyst*, 1955, **80**, 906) for the determination of three iron chelates of the polyaminepolycarboxylic type has been successfully applied to three other chelates, *viz*, the Fe^{III} chelates of NN′-di(hydroxyethyl)ethylenediaminediacetic acid (Fe - HEEDDA), *cyclo*hexane-1:2-diaminetetra-acetic acid (Fe - CDTA) and ethanediamine-NN′-bis-(*o*-hydroxyphenylacetic acid) (Fe - EDHPA). The spectra of these chelates vary greatly with the pH of the soln. For Fe - HEEDDA, soln. of pH 2·8 ± 0·1 had an absorption peak at 250 mμ and soln. of Fe - CDTA had similar spectra for the pH range 2 to 9. Soln. of Fe - EDHPA within the range pH 4 to pH 10 all had a peak at 485 mμ, a peak at ≃280 mμ being unsuitable for quant. determination owing to interference from EDHPA. A. O. JONES

103. **High-frequency [magnetic permeability] determination of ferro-magnetic metals.** F. J. Schmidt (Nickel Processing Corp., Nicaro, Oriente, Cuba). *Anal. Chem.*, 1957, **29** (7), 1060–1062.—The magnetic permeability is determined by means of its effect on the induction of a surrounding coil forming part of a described tuned oscillation circuit. The effect is almost linear, but results are obtained from empirical calibrations of chemically analysed samples. The precision is said to be ±0·38% for reduced NiO, and the time consumption is small.
D. A. PANTONY

104. **Sampling methods for the determination of hydrogen in steel.** L. Bjerkerud (Surahammars Bruk AB, Sweden). *Jernkontor. Ann.*, 1957, **141** (2), 94–99.—Various methods of sampling and casting are described, steel or copper moulds being used; copper moulds are preferred. The hydrogen content of several samples of steel is given, and analytical results on ground and unground test-pieces are compared. If there is a visible film of oxide on the sample the grinding should be carried out immediately before the analysis. The sample should be weighed, washed in CCl_4, dried and transferred to the apparatus. P. HAAS

105. **Apparatus for the determination of hydrogen in steel.** L. Bjerkerud (Surahammars Bruk AB, Sweden). *Jernkontor. Ann.*, 1957, **141** (2), 90–94.—A simplified apparatus for the determination of hydrogen in steel is described and illustrated. The results obtained agree closely with those obtained by the older methods in which the gas is passed through a palladium tube. Reference is made to the paper by Newell on the estimation of hydrogen (*J. Iron St. Inst.*, 1940, **141**, 243). P. HAAS

106. **Determination of small quantities of aluminium in steel. II.** A. Neuberger and E. Schöffmann (Niederrheinischen Hütte A.-G., Duisburg, Germany). *Arch. Eisenhüttenw.*, 1957, **28** (2), 65–66.—It is shown that the 25% loss of Al in the sodium hydroxide procedure (*cf.* Werz and Neuberger, *Anal. Abstr.*, 1956, **3**, 3640) can be reduced to 3% by using the magnetic concentration method (*cf.* Zörner *et al.*, *Tech. Mitt., Rheinhausen*, 1955, 237). The sample is dissolved in dil. HCl, and $FeCl_3$ is added, followed by aq. NH_3. The soln. is boiled and the flask is placed on a magnet, and the ppt. thus becomes firmly deposited on the walls of the flask. The liquid is decanted and the ppt. dissolved in an HCl - HNO_3 mixture. Aluminium is determined photometrically in this soln. after boiling with NaOH and H_2O_2. G. BURGER

107. **Colorimetric determination of combined carbon in low alloy constructional steels.** L. V. Zaslavskaya and N. M. Popova. *Zavod. Lab.*, 1957, **23** (6), 656–657.—The normal method for the colorimetric determination of C in steel is modified by dissolving in HNO_3 not the metal but the carbide residue obtained by anodic dissolution of the steel. To increase the sensitivity further, H_3PO_4 is added to remove the colour due to Fe. G. S. SMITH

108. **Polarographic determination of tin and antimony in iron and steel.** L. Brháček (VŽKG, Ostrava, Czechoslovakia). *Hutn. List.*, 1957, **12** (2), 140–141.—A method for the rapid determination of Sn and Sb in iron and steel that are sol. in HNO_3 is described. This method was applied satisfactorily to the determination of both these elements in copper. J. Böswart

109. **Absorptiometric determination of vanadium in steel.** P. H. Scholes [Metallurgy (General) Div. Lab., B.I.S.R.A., Sheffield, England]. *Analyst*, 1957, **82**, 525–528.—Two colour reactions for the determination of V in steel have been examined, the reagents being **3**:3′-dimethylnaphthidine (**I**) (Milner *et al.*, *Brit. Abstr. C*, 1949, 379) and tungstophosphoric acid (**II**) (Lennard, *Analyst*, 1949, **74**, 253). The reaction with **I**, although extremely sensitive, was somewhat erratic under the conditions investigated and was considered an unsuitable basis of a precise macro-analytical method. The reaction with **II** is less sensitive, but more reliable. The sample (0·5 g) is dissolved in a H_2SO_4 - H_3PO_4 mixture and fumed with HNO_3. An aliquot of the diluted digest is titrated with 0·01 *N* $KMnO_4$ until the pink colour persists, and 2 drops are added in excess. The liquid is treated with specified amounts of dil. H_3PO_4 and $Na_2WO_4.2H_2O$, adjusted to 50 ml, boiled, cooled and re-adjusted to vol. Its extinction is then measured in an absorptiometer against an equal aliquot of the sample soln. treated similarly but with omission of the Na_2WO_4. The result is corrected for the extinction of a vanadium-free iron blank similarly treated and is referred to a calibration graph. The method offers a fivefold increase in sensitivity over the classical method with H_2O_2. A. O. Jones

110. **Effect of chromium in the bismuthate method for the determination of manganese [in iron alloys].** W. E. Clarke and W. B. Shaw (B.C.I.R.A., Alvechurch, Birmingham). *J. Res. Brit. Cast Iron Ass.*, 1957, **6** (10), 512–518.—It is shown that Cr in amounts up to 5% does not interfere in the bismuthate method provided that sufficient excess of freshly prepared saturated SO_2 soln. is added to reduce any chromate formed during the hot oxidation. S.C.I. Abstr.

111. **Determination of cobalt in ferrous alloys using a chromatographic separation.** D. G. Stern. *Metallurgia, Manchr*, 1957, **55**, 207–208.—The ferrous alloy is dissolved in HCl and oxidised wih H_2O_2. Cobalt is separated chromatographically from interfering elements on a cellulose column, with a mixture of ethyl methyl ketone and HCl as eluent. It is finally determined gravimetrically by pptn. with 1-nitroso-2-naphthol. S.C.I. Abstr.

112. **Rapid photometric determination of nickel with dimethylglyoxime.** L. Doubek (Spojené strojírny a slévárny B. Šmerala, Brno, Czechoslovakia). *Hutn. List.*, 1957, **12** (5), 430–432.—The determination is based on the colour measurement of the red complex of Ni with dimethylglyoxime after oxidation with iodine. *Procedure*—Dissolve the sample (0·25 to 0·5 g) in aqua regia (20 to 40 ml), and to 10 ml add 0·32 *N* iodine (10 ml) and dimethylglyoxime in a mixture of NaOH and tartaric acid (25 ml). After 8 min. measure the extinction against a blank. J. Böswart

113. **A new photometric determination of nickel with rubeanic acid [dithio-oxamide].** L. Farády and A. Jánosi (Dept. Anal. Chem., Univ. Chem. Ind., Veszprém, Hungary). *Magyar Kém. Foly.*, 1957, **63** (1), 19–23.—The blue complex of Ni and dithio-oxamide (**I**) is suitable for photometric determination. The absorption has a max. at pH 8·2. In the presence of pyridine, a purple complex is formed, which can be determined similarly, the optimum pH being 8·6 to 9·6. *Procedure*—To the acid nickel soln. add phenolphthalein indicator (2 drops) and 1% gelatin soln. (1 ml); make the soln. alkaline with approx. *N* aq. NH_3 and then very slightly acid with approx. 0·2 *N* HCl. Add 5% Na acetate buffer (1 ml) and 0·2% ethanolic **I** and dilute to 50 ml with H_2O. Wait for 30 min. to 5 hr. before determining the extinction on a Pulfrich photometer, with an S57 filter and a 20-mm cell. The Beer - Lambert law is obeyed for 0·5 to 8·0 mg of Ni per litre. The average relative error is ±1%. Alternatively, add to the nickel soln. (neutral to litmus) 0·1 *N* aq. NH_3 (1 ml), 5% Na acetate (1 ml), 1% gelatin soln. (protective colloid) (1 ml), pyridine (3 ml) and 0·2% ethanolic **I** (1 ml), and dilute to 50 ml with water. Wait for 30 min. and determine the extinction as above, but with an S53 filter. The Beer - Lambert law is obeyed for 1 to 8 mg of Ni per litre. The accuracy is ±1·3%. Copper, cobalt and ferric ions interfere in both methods; their removal is described. In alloys and steels the relative error is ±6%. A. G. Peto

114. **Use of organic compounds in the analysis of the platinum metals and gold. III. The use of phenothiazine for determining platinum in alloys.** M. S. Usova and N. F. Gaeva. *Trudy Ural'sk. Politekh. Inst.*, 1956, (57), 201–206; *Ref. Zhur., Khim.*, 1957, Abstr. No. 30,932.—A study is made of the ability of phenothiazine to precipitate separate platinum metals (Pt, Pd, Rh and Ir) for the purposes of qual. analysis, and a method is evolved for the determination of Pt in pure salts of Pt, in artificial mixtures and in Ag - Pt alloys, by pptn. of Pt with phenothiazine and ignition of the ppt. to metallic Pt. C. D. Kopkin

See also Abstracts 3, 10, 178, 300, 306, 318, 319, 337.

3.—ORGANIC ANALYSIS

115. **Semi-micro determination of chlorine in highly-chlorinated compounds.** J. Estevan and J. Serra (Org. Chem. Dept., Inst. "Alonso Barba," Univ. Barcelona, Spain). *An. Real Soc. Esp. Fis. Quim., B*, 1957, **53** (3), 233–236.—The standard method of Belcher and Godbert for the semi-micro determination of Cl is modified for use with highly-chlorinated hydrocarbons, etc., by increasing the temp. of combustion to 950°, and by using an additional furnace to keep the $BaCO_3$ at ≃650°. Correct values are obtained for a range of 14 compounds containing 55·6 to 77·1% of Cl. C. A. Finch

116. **Silicon determination in low-boiling organosilicon compounds by acid digestion.** B. Smith (Inst. Org. Kemi, Chalmers Tek. Högskola, Göteborg, Sweden). *Acta Chem. Scand.*, 1957, **11** (3), 579–580 (in English).—Compared with the Kjeldahl method, the quicker crucible method of acid digestion was unsuitable for low-boiling compounds because of evaporation losses during the early stage

of heating. By using a system of two crucibles, a stronger oxidising acid mixture (equal vol. of 60% fuming H_2SO_4 and of fuming HNO_3), ice-cooled, and by introducing the substance well below the acid from a sealed ampoule, losses are reduced to a minimum. Any overflow due to frothing from the inner 10-ml reaction crucible is caught in the outer 50-ml crucible. Good correspondence with the theoretical content of Si was obtained, even for compounds that react violently with the acid mixture. F. J. M.

117. **Application of anhydrous alkalimetry to the determination of the epoxy group.** C. Hennart and E. Merlin. *Chim. Anal.*, 1957, **39** (7), 269–271.—The method given, which is applicable whether the epoxide contains an aromatic radical or not, obviates the need for a correction for the presence of acids or acidic compounds in the sample. *Procedure*—Place the sample (≡ 0·002 g of epoxy oxygen) in a small glass cup inside a conical flask having a ground-glass neck. Add 25 ml of 0·2 *N* anhyd. HCl (dissolved in a mixture of CCl_4 and di*iso*propyl ether), and keep the stoppered flask at ≃20° for ≮6 hr. Then titrate the excess of HCl with a soln. of 0·2 *N* Na acetate in anhyd. acetic acid, with methyl violet as indicator. The values calculated for the O of the epoxide ring agree closely with those obtained by the Swern method (*cf. Brit. Abstr. C*, 1947, 245). W. J. Baker

118. **Colorimetric assay for reaction of sulphydryl groups with organic mercurials.** I. Fridovich and P. Handler (Duke Univ. Sch. of Med., Durham, N.C., U.S.A.). *Anal. Chem.*, 1957, **29** (8), 1219–1220.—Sulphydryl compounds can be determined colorimetrically by reaction with excess of *p*-chloromercuribenzoate. The excess is measured by its bleaching effect on dithizone at 625 mμ. Linear response was obtained over the range 0·01 to 0·05 μmole of cysteine, and 0·03 μmole of cysteine yielded an absorbance change of 0·155. Triplicate determinations agreed to within 3%. G. P. Cook

119. **Micro-amperometric titration of sulphydryl groups.** S. Levine (Gen. Engng Lab., American Machine & Foundry Co.). *Instrum. and Automation*, 1957, **30** (5), 883.—The sample, containing as little as 10^{-6} milli-equiv. of –SH, is contained in a 2-ml sintered-glass funnel. Electrical contact is provided by means of an agar gel in 0·1 *M* KNO_3 in the stem of the funnel. The other electrode is a vibrating platinum tip. The titrant is introduced by means of an ultra-micro burette. G. Skirrow

120. **Thermomagnetic and infra-red method for gas analysis in the manufacture of acetylene from methane.** A. Németh and C. Cîrlogan (Inst. Mag. Cercetări Pentru Petrol şi Gaze Naturale, Veszprem). *Rev. Chim., Bucharest*, 1957, **8** (2), 93–99.—Apparatuses have been combined in series for the determination of acetylene by its infra-red absorption, and the determination of oxygen based on its paramagnetic quality. A precision of 0·05% is claimed over the range 0 to 3% of acetylene, and of 0·1% for the range 3 to 10%; for oxygen the precision is 0·01% over the range 0 to 1%. H. Sher

121. **Micro-determination of density of liquid hydrocarbons by rising-drop method.** G. Nettesheim (Inst. Chem. Technol., Tech. Hochschule, Aachen). *Erdöl u. Kohle*, 1957, **10** (2), 73–74.—The speed of ascent or descent of a drop through a liquid of different d is proportional to the density difference Δd. A droplet of the hydrocarbon of fixed volume is discharged into the lower end of a water column (tube 150 cm by 5 cm bore) from the capillary leg (0·25 mm bore) of a U-shaped micropipette by moving a mercury thread between two fixed points 7 cm apart on the capillary by means of a screw-threaded piston (*e.g.*, a fine file wrapped with leather) working in the outer wider (2·5 mm) leg of the U through a connecting tap. The ascent of the droplet between marks 100 cm apart on the water tube is timed to 0·01 sec. Duplicate timings agree within 0·5%. The drop-forming capillary nozzle must be drawn out fine and may be treated with an anti-foaming preparation. Liquids of known d are used to construct a calibration curve with time (t sec.) as ordinate and d as abscissa. Alternatively, $1/t$ may be plotted against Δd ($= d \sim d_{20}H_2O$), the graph being then nearly rectilinear as required for laminar motion and applicable to liquids denser than water since it involves only a density difference. The apparatus can be adapted to time the fall of the denser droplets. A. R. Pearson

122. **Colorimetric method for determining minute quantities of chloroform in carbon tetrachloride.** C. D. Hildebrecht (Research Center, Diamond Alkali Co., Painesville, Ohio, U.S.A.). *Anal. Chem.*, 1957, **29** (7), 1037–1039.—The method, suitable for 10 to 900 p.p.m., depends on the measurement of the red colour developed by the reaction of pyridine and NaOH with $CHCl_3$ in the presence of CCl_4. Into a clean dry 100-ml cylinder place water (5·0 ml), the sample (1·0 ml) and 10% NaOH soln. (5 drops). Add pyridine (15 ml), mix, and immerse in a boiling-water bath for 3 min. ±5 sec. Cool in water (15° to 20°) for 7 min., dilute with methanol to 100 ml, mix thoroughly and allow to stand for 10 min., then measure the extinction against water, with a green (525 mμ) filter. Results are referred to a standard curve. The times stated must be adhered to as closely as possible. Good reproducibility and precision are shown in the recovery of known amounts of $CHCl_3$ from 25 to 600 p.p.m. Blanks should be run on all reagents and unsatisfactory ones purified by redistillation. Of 12 chlorinated compounds examined, only 1:1:1:2- and 1:1:2:2-tetrachloroethane, pentachloroethane and 1:1-dichloroethylene react in the same way as $CHCl_3$, but these should not occur in significant amounts in CCl_4. Carbon disulphide in concn. >20 p.p.m. interferes, but can be recognised by causing an immediate yellowing of the mixture before immersion in boiling water. R. E. Essery

123. **Determination of vicinal glycols by oxidation with periodate in non-aqueous media.** R. J. B. Reddaway (Roche Products Ltd., Welwyn Garden City, England). *Analyst*, 1957, **82**, 506–511.—The sample (140 to 160 μ-equiv.) of the glycol in ethanolic soln. (5 ml) is treated with exactly 5 ml of a non-aq. periodate reagent (prep. described) in a 100-ml flask and is set aside in the dark for 75 min. As a blank determination, 5 ml of ethanol is treated similarly. The liquids are then treated first with 10 ml of *M* $NaHCO_3$, exactly 5 ml of 0·1 *N* sodium arsenite and 2 ml of 20% KI soln. After 15 min. the liquids are titrated with 0·05 *N* iodine soln. The method is accurate and precise for batyl alcohol and *cyclo*hexane-1:2-diol. A. O. Jones

124. **Colorimetric determination of propane-1:2-diol and related compounds.** L. R. Jones and J. A. Riddick (Commercial Solvents Corp., Terre Haute,

Ind., U.S.A.). *Anal. Chem.*, 1957, **29** (8), 1214–1216.—Propane-1:2-diol is dehydrated in conc. H_2SO_4 to give a mixture of allyl alcohol and the enolic form of propionaldehyde, which forms a violet complex with ninhydrin in conc. H_2SO_4 soln. The colour obeys Beer's law in the range 5 to 50 μg at a wavelength of 595 mμ. The accuracy is within ±2% and the precision is within ±1%. The colour is specific for propane-1:2-diol and its polymers in mixtures of diols. G. P. Cook

125. **A micro-scale classification and determination of sugars using the phloroglucinol reaction.** N. O. Lindh (Inst. of Zoophysiology, Univ. of Lund, Sweden). *Ark. Kemi*, 1957, **10** (6), 569–576 (in English).—*Procedure*—Duplicate samples of the sugar soln. (1 vol.) are each mixed with 8 vol. of soln. *A* [0·1% $FeCl_3$ in conc. HCl (1 vol.) and glacial acetic acid (6 vol.)]; the tubes are closed and kept at 100° for 50 min., then cooled to room temp.; 1 vol. of soln. *B* [0·25% phloroglucinol in conc. HCl (1 vol.), glacial acetic acid (2 vol.), and water (1 vol.)] is added to each tube. The tubes are closed, shaken until the contents are homogeneous and then set aside at room temp. for 20 min. One tube is then heated for 4 min. and the other for 40 min., and both are immediately cooled in ice-water. The colour is measured, in a spectrophotometer, against a blank soln. All sugars give a yellow colour (max. extinction 440 to 460 mμ); this colour soon fades but the heat treatment gives a permanent colour. After heating for 4 min., pentoses give a colour which has max. extinction at 620 to 630 mμ, aldohexoses at 422 mμ and uronic acids at 570 mμ. After 40 min., the change in wavelength is characteristic for individual sugars. C. A. Slater

126. **Determination of pentosans. I. A u.v. spectrophotometric method for determination of furfuraldehyde and 5-hydroxymethylfurfuraldehyde in distillates after Tollens distillation.** P. O. Bethge (Swedish Forest Products Res. Lab., Stockholm). *Svensk Papp.-Tidn.*, 1956, **59**, 372–376.—A procedure is outlined for the determination of pentosans by spectrophotometric observations after conversion into furfuraldehyde (**I**). The presence of hydroxymethylfurfuraldehyde (**II**) involves a separation by the Craig counter-current technique. The primary distillation is carried out according to the method of the Analytical Committee of the Cellulose Industries Central Lab. (*Svensk Papp.-Tidn.*, 1941, **44**, 267), with a 2-g sample. For the separation of **I** and **II**, the distillate is diluted to 250 ml with HCl (13·15%) and an aliquot (25 ml) is used in the apparatus with $CHCl_3$ as the stationary phase and HCl (13·15%) as the mobile phase. The experiment is run until the mobile phase reaches the 25th tube, when it is found that **I** is concentrated in the tubes 1 to 7, and **II** in the tubes 15 to 23. The contents of these two groups of tubes are made up to 100 ml with ethanol and the extinctions of the two resulting soln. are determined with a Beckman DU spectrophotometer at 277·5 mμ. From a knowledge of the specific extinctions of **I** and **II**, the amounts of these compounds originally present can be calculated. Experiments showed a recovery of 90 to 109%.

II. The use of barbituric acid for the determination of furfuraldehyde. P. O. Bethge and R.-M. Persson. *Ibid.*, 1956, **59**, 535–539.—A critical review is given of the method for determination of **I** in distillates obtained with the Tollens procedure. Pptn. with barbituric acid is of little value as the reaction is affected by many variable factors, among which are the amount of **I** to be estimated, the acidity of the distillate, the temp. of the reaction mixture, the purity and concentration of the barbituric acid reagent, and the presence of interfering substances. D. G. Forbes

127. **Dextran. II. Determination of the mean molecular weight of partially degraded dextran by an iodimetric method.** L. Lacko and J. Málek (Inst. Haematol. and Transfusion, Prague). *Chem. Listy*, 1957, **51** (1), 47–50.—Values for mean mol. wt. of partially degraded dextran in infusion soln. were determined by a method depending on the oxidation of the aldehydic groups with excess of iodine in NaOH soln. and titrating the excess of iodine with $Na_2S_2O_3$. Dextrans degraded by various strains of *Leuconostoc mesenteroides* gave values from 22,400 to 46,800. Results by osmometric and reduction methods varied by up to 7% and 27%, respectively. J. Zýka

128. **Study of the absorption spectra of the 2:4-dinitrophenylhydrazones of carbonyl compounds.** I. N. Nazarov, L. A. Kazitsýna and I. I. Zaretskaya (Chem. Inst., Acad. Sci., Armenian SSR). *Zhur. Obshch. Khim.*, 1957, **27** (3), 606–623.—The u.v. absorption spectra of >100 2:4-dinitrophenylhydrazone derivatives in *iso*octane and heptane soln. are studied. Starting with the formaldehyde compound which has a max. absorption at 330 mμ, it is shown that the position of the max. for other carbonyl compounds is determined by the degree of substitution of H by alkyl at the carbonyl group, conjugation and β-substitution in a vinyl chain. The introduction of the first conjugated double-bond into formaldehyde causes a shift (increase) in the max. of 17 mμ, and successive conjugated double-bonds cause a shift of 9 mμ. These and other rules enable the structure of aldehydes and ketones to be assessed spectroscopically. G. S. Smith

129. **Detection and determination of acetic acid by the Duclaux method.** J. Soleil and G. Hirt. *Compt. Rend. Acad. Sci., Paris*, 1957, **244** (15), 2059–2061.—The Duclaux method of qual. and quant. determination of volatile acids was tested with 1·25% and 2% aq. solutions of acetic acid and found to give inaccurate and unreliable results. S.C.I. Abstr.

130. **Simultaneous determination of diethyl esters of monoethylmalonic and diethylmalonic acids in commercial diethyl diethylmalonate.** Z. Bellen and B. Sękowska (Anal. Dept., Inst. of General Chem., Warsaw). *Chem. Anal., Warsaw*, 1957, **2** (1), 35–43.—The method depends on the different rates of hydrolysis of diethyl malonate (**I**), diethyl monoethylmalonate (**II**) and diethyl diethylmalonate (**III**) by alkali. **I** (in 1 g of **III**) is hydrolysed by shaking for 5 min. with a 25% aq. KOH soln. (5 ml) and the excess of KOH is determined by titration with 0·5 *N* HCl, with phenolphthalein indicator. **I** and **II** (in 1 g of **III**) are hydrolysed by shaking with *N* KOH soln. in *n*-butanol (10 ml) for 5 min. at room temp. and heating in a boiling-water bath for 2 min. The soln. is cooled, acetone (5 ml) is added and the pptd. potassium salts of the two acids are filtered off, washed with acetone - *n*-butanol (1:1), dried at 150°, and weighed. The total esters (in 1 g of **III**) are determined by hydrolysis with 2 *N* KOH in water - ethanol (1:3) (25 ml), the mixture is heated in a boiling-water bath for 4 hr. and the excess of KOH is titrated with 0·5 *N* HCl. The accuracy of the method is within ±2%. K. F. Sporek

131. **Determination of aliphatic primary amino nitrogen compounds by reaction with pentane-2:4-dione.** F. E. Critchfield and J. B. Johnson (Union Carbide Chem. Co., S. Charleston, W. Va., U.S.A.). *Anal. Chem.*, 1957, **29** (8), 1174–1176.—A measured excess of pentane-2:4-dione reacts with a primary amine to form an imine. The excess of reagent is titrated as an acid with Na methoxide in pyridine, with thymolphthalein as indicator. Most acids and bases with ionisation constants greater than 1×10^{-2}, large quantities of water, and some secondary alcohol amines interfere. Data are listed for the determination of 23 primary amines and show that an accuracy within $\pm 0{\cdot}1\%$ can be attained in the presence of secondary amines. The method is also applicable to the determination of primary alcohol amines, ethyleneamines, amino acids, aliphatic amines and NH_3. G. P. Cook

132. **Detection of small amounts of amines.** J. Mlodecka and A. Hojnacka (Inst. Gen. Chem., Warsaw). *Chem. Anal., Warsaw*, 1957, **2** (2), 193–194.—The amine is acidified with HCl, and NH_3 is liberated by the addition of CaO. The fumes form a stain on a filter-paper saturated with $Mn(NO_3)_2$ - $AgNO_3$ indicator. The black or grey stain turns blue with one drop of benzidine. H. Dmowska

133. **Modification of the diacetyl determination of urea.** R. L. LeMar and D. Bootzin (Rock Island Arsenal Lab., Ill., U.S.A.). *Anal. Chem.*, 1957, **29** (8), 1233–1234.—The photometric method for the determination of urea, with diacetyl as the colour reagent, was modified for the analysis of volatile corrosion-inhibitor materials. Changes were made in the volume of reacting soln., reaction time, cooling time, and concn. of the diacetyl reagent. The concn. range was 75 to 225 μg of urea, and an average coefficient of variation of $0{\cdot}917\%$ was obtained from triplicate determinations. Sodium nitrite interfered, but a correction can be applied. G. P. Cook

134. **Titration in non-aqueous medium. III. Determination of guanidine nitrate in a technical product.** J. Minczewski and J. Mlodecka (Phys. Chem. Inst., Polish Acad. Sci., Warsaw). *Chem. Anal., Warsaw*, 1957, **2** (2), 176–182.—Guanidine and NH_4^+ are titrated together in glacial acetic acid with $HClO_4$, and NO_3^- with Na methoxide in methanol. Both titrations are carried out potentiometrically, with a silver chloride electrode and a glass electrode for the $HClO_4$ titration, and a silver chloride electrode and a molybdenum or antimony electrode for the Na methoxide titration. The impurities usually present in technical guanidine do not interfere. The time of one determination is approximately 1·5 hr. The error is approximately $\pm 1\%$. H. Dmowska

135. **Determination of small amounts of xanthate in aqueous solutions by potentiometric titration.** G. A. Osolodkov (Leningrad Mining Inst.). *Zavod. Lab.*, 1957, **23** (6), 764.—The method of Plaksin *et al.* (*Anal. Abstr.*, 1956, **3**, 2134) requires modification in the presence of much alkali. It is recommended that the electrode and titrant be replaced by Hg and $HgCl_2$, respectively, and that the alkalinity of the soln. be reduced by addition of $MgSO_4$. G. S. Smith

136. **Mineralisation of organic materials. Tetraethyl-lead. III.** L. García Escolar and E. Niño Martínez (Valladolid Univ., Spain). *An. Real Soc. Esp. Fis. Quim., B*, 1957, **53** (2), 161–170.—Lead in "ethyl fluid" is pptd. instantaneously, as the dihalide, by a CCl_4 soln. of Cl (**I**) or ICl_3 (**II**). Dilute a known weight ($\simeq 0{\cdot}3$ g) of "ethyl fluid" with 10 ml of CCl_4, and add **I** or **II** in CCl_4 soln., dropwise, until there is no further pptn. Filter, wash the ppt. with CCl_4, and dry at 100° to eliminate CCl_4. Dissolve the ppt. in ammonium acetate or disodium phosphate and determine the Pb. G. F. Penny

137. **Quantitative determination of small quantities of benzene.** V. D. Bogatskiĭ and A. V. Mozharovskaya. *Tr. Odessk. Univ. Ser. Khim.*, 1956, **146** (5), 107–109; *Ref. Zhur., Khim.*, 1957, Abstr. No. 27,243.—Benzene (0·1 to 0·001 g) is treated with phthalic anhydride at 75° in the presence of dry $AlCl_3$ to form *o*-benzoylbenzoic acid which is converted into anthraquinone by heating with H_2SO_4 at 150°. This is reduced with NaOH and Zn forming the red colour of 9:10-anthradiol. L. Herdan

138. **Determination of small amounts of *o*-phenylphenol.** D. Harvey and G. E. Penketh (Res. Dept., I.C.I. Ltd., Billingham, Co. Durham, England). *Analyst*, 1957, **82**, 498–503.—Suitable methods for extraction of the fungicide *o*-phenylphenol depend on the nature of the material, and typical examples are given, the extracting solvents being *cyclo*hexane and NaOH soln. With some materials, preliminary steam-distillation is necessary. The final soln. should contain 5 to 25 μg per ml. The determination may be made spectrophotometrically at 284 and 295 mμ for *cyclo*hexane soln., or at 300 and 320 mμ for soln. in NaOH, the double readings serving to detect interference. Determinations may also be made by comparing the fluorescence in u.v. light of the sample in NaOH soln. with that of standard soln. In a third colorimetric method the cherry-red colour produced by Brentamine fast red GG with the sample is compared visually with standards, or is measured spectrophotometrically at 494 mμ. The last method is the only one applicable to impregnated timber. A. O. Jones

139. **Significance of pH in determination of vanillin by ultra-violet absorption.** D. T. Englis and L. A. Wollermann (Univ. Ill., Urbana, U.S.A.). *Anal. Chem.*, 1957, **29** (8), 1151–1153.—The u.v. absorption curves for vanillin under acid and basic conditions have been studied, and results similar to those observed by Lemon (*J. Amer. Chem. Soc.*, 1947, **69**, 2998) for alcoholic soln. were obtained. Adjustment of pH to values above 9·6 and below 5·0 is recommended for quant. absorptiometric analysis of the basic and acid forms, respectively. Three well-defined isosbestic points near 238, 259 and 316 mμ are not dependent on pH, and suggest possible analytical application. The pK value of vanillin was found to be 7·3, and vanillin at pH 10·7 was found to be stable for over a month. G. P. Cook

140. **Analytical examination of mixtures of adipic, sebacic and phthalic acids.** M. Clasper and J. Haslam (I.C.I. Ltd., Plastics Div., Welwyn Gdn. City, England). *J. Appl. Chem.*, 1957, **7** (6), 328–332.—Under controlled conditions of pH, sebacic and adipic acids can be qual. separated from mixtures with phthalic acid. The acids are eluted successively with *n*-butanol - $CHCl_3$ mixtures of given concn. from a chromatographic column of silicic acid (100 mesh). An accuracy of $\pm 2\%$ is

claimed and the method is applicable to the analysis of some other aliphatic homologues. *Procedure*—Titrate *N* HCl (25 ml) with *M* Na citrate soln. to a pH of 4·3 and add 10 ml of this buffer soln. to 10 g of silicic acid. Add *n*-butanol - $CHCl_3$ (2% v/v) (40 ml), stir to form a slurry and fill a chromatographic column. Place a disc of filter-paper on top of the column, which must be kept moist with solvent. Dissolve the acid sample (0·5 g) in warm *tert.*-amyl alcohol (10 ml) and dilute with $CHCl_3$ to 50 ml. Transfer 5 ml of this soln. on to the column, add *n*-butanol - $CHCl_3$ (2%) (2 ml) and elute with a further quantity (25 ml). Titrate the eluate (sebacic acid fraction) with 0·05 *N* NaOH, with *m*-cresol purple as indicator, until a purple colour persists for ± 5 sec. Further elute the column with *n*-butanol - $CHCl_3$ (15%) (70 ml) and titrate as before (adipic acid fraction). Blank experiments should be run concurrently through a second column.

D. G. FORBES

141. **Polarographic and oscillographic differentiation of esters of the phthalic acids.** A. Ryvolová (Polarographic Inst., Acad. Sci., Prague, Czechoslovakia). *Chem. Listy*, 1957, **51** (6), 1201–1202.—Dimethyl phthalate (**I**), dimethyl *iso*phthalate (**II**), dimethyl terephthalate (**III**) and trimethyl trimesate (**IV**) yield well-developed polarographic waves in soln. of pH 9 to 10. The differentiation of **I** and **II** (or **III**), and **II** in the presence of **IV**, is possible. The Britton - Robinson buffer soln. and 0·05 *N* borate are good supporting electrolytes. The $E_{\frac{1}{2}}$ values for **I**, **II**, **III** and **IV** are given.

J. ZÝKA

142. **Polarographic determination of *m*-dinitrobenzene in mixtures with 1-chloro-2:4-dinitrobenzene.** J. Roubal and J. Zdražil (Inst. Hyg. and Ind. Diseases, Prague, Czechoslovakia). *Chem. Listy*, 1957, **51** (6), 1199–1200.—*Procedure*—Prepare a soln. of the sample in H_2O or in dil. ethanol (the concn. of ethanol must not exceed 5%) (200 mg in 1 litre), transfer to a 500-ml flask and hydrolyse with Na_2CO_3 soln. (0·5%) at 95° (60 min.). Extract a 100-ml portion with ether (3 × 50 ml). Evaporate the combined extracts on a water bath, dissolve the residue in a small portion of ethanol in H_2O and add Na_2CO_3 soln. till the concn., after the vol. has been adjusted to 100 ml, is 0·5%. Determine the *m*-dinitrobenzene polarographically. The alkaline aq. soln. (after the extraction) can be polarographed directly and the 2:4-dinitrophenol thus determined. 1-Chloro-2:4-dinitrobenzene can be calculated from the value for 2:4-dinitrophenol. Its content can also be calculated by polarographing the resulting chlorides in 0·1 *N* HNO_3. The error does not exceed $\pm 5\%$.

J. ZÝKA

143. **Determination of 4-aminodiphenyl in technical diphenylamine.** H. E. Stagg and R. H. Reed (I.C.I. Ltd., Hexagon House, Blackley, Manchester, England). *Analyst*, 1957, **82**, 503–506.—In the method described, the 4-aminodiphenyl is extracted with dil. HCl, diazotised and coupled with R-salt (disodium 2-naphthol-3:6-disulphonate). The coloured soln. is then chromatographed on a pad of six 32-cm Whatman No. 1 filter-papers. The paper is eluted with NaOH soln. according to a specified technique and the extinction of the eluate is measured in a Spekker absorptiometer with Chance OB 1 (No. 7) glass filters. With the tarry residues obtained by distillation of technical diphenylamine it is often advisable to remove the volatile matter from the tar by distillation at low pressure and to test the fractions separately. Typical samples of diphenylamine contained 0·007 to 0·025% of 4-aminodiphenyl.

A. O. JONES

144. **New analytical method for diphenylthiourea.** F. Hernández-Gutierrez (Inst. Anal. Chem., Fac. Pharm., Barcelona, Spain). *An. Real Soc. Esp. Fis. Quim., B*, 1957, **53** (3), 211–216.—A new test for diphenylthiourea, based on the reaction with alkaline mercuric potassium iodide, which gives a yellow complex, is described. The limit of the sensitivity of the reaction, which follows Beer's law up to 150 μg per ml, does not exceed 10 μg per ml.

C. A. FINCH

145. **X-ray diffraction powder data for some copper N-alkylsalicylaldimine chelates.** R. G. Charles and W. D. Johnston (Westinghouse Res. Lab., Churchill Borough, Pittsburgh, Pa., U.S.A.). *Anal. Chem.*, 1957, **29** (8), 1145–1146.—Data are presented for 11 copper chelates derived from straight-chain primary amines and permit identification of unsubstituted straight-chain primary amines containing from 1 to 14 C atoms.

K. A. PROCTOR

146. ***p*-Phenylazophenacyl esters. Rates of movement relative to *p*-phenylazophenacyl bromide on silicic acid and identification by paper partition chromatography.** R. E. Kepner, A. D. Webb, R. L. King and A. D. Bond (Univ. Calif., Davis, U.S.A.). *Anal. Chem.*, 1957, **29** (8), 1162–1164.—Small quantities of *p*-phenylazophenacyl derivatives of organic acids are identified by comparing their rates of travel on silicic acid columns with that of *p*-phenylazophenacyl bromide. Data for $\simeq 40$ derivatives are listed. A paper partition chromatographic system, with 2-phenoxyethanol-impregnated paper as the stationary phase and *n*-heptane saturated with 2-phenoxyethanol as the mobile phase, is also described for the identification of *p*-phenylazophenacyl derivatives, and data for $\simeq 30$ of these are presented.

G. P. COOK

147. **Determination of 1-naphthol-3:6:8-trisulphonic acid in the presence of 1:8-dihydroxynaphthalene-3:6-disulphonic acid (chromotropic acid).** A. Spiliadis, D. Bretcanu and E. Badică. *Rev. Chim., Bucharest*, 1957, **8** (4), 296–301.—Contrary to expectation, both 1-naphthol-3:6:8-trisulphonic acid (**I**) and chromotropic acid (**II**) coupled with one mol. of diazo compounds, of which *p*-nitrophenyldiazonium chloride (**III**) gave the most reliable results, at pH 6. On the other hand, at pH 12 to 12·5 **I** absorbed 2 atoms and **II** 4 atoms of iodine. It is therefore possible by carrying out two titrations (*i*) with **III** in the usual manner and (*ii*) by adding 50 ml of 0·1 *N* iodine to a soln. containing 2% of NaOH and, after shaking for 15 min., adjusting to pH 6 with conc. HCl and titrating the excess of iodine, to calculate the proportions of **I** and **II**. The maximum error is $\pm 2\%$.

H. SHER

148. **Improved method for the analysis of pyridine bases.** G. Lindner. *Ark. Kemi*, 1957, **10** (5), 483–495.—The method of Vignes and Chervet (*Brit. Abstr. C*, 1952, 304) for the separation and identification of pyridine bases has been improved, and several of the $CuCl_2$ complexes of the pyridine bases have been prepared as pure compounds. *Procedure*—Active Al_2O_3 (Brockmann standardised) (250 g) is stirred into a soln. of 20 g of anhyd. $CuCl_2$ in 125 g of anhyd. methanol, and, after

being dried at 50° to 60° for 8 hr., affords a homogeneous free-flowing powder. For analyses, this powder is filled into tubes (3 mm × 30 cm) and 0·2 to 1·0 ml of the pyridine-base sample dissolved in 30 ml of light petroleum is transferred to the column. After passage of the sample through the column, the quantities of the bases present are estimated from the lengths of the coloured zones and the mol.-wt. ratios of the different complexes. Characteristic colours are given for the copper complexes of pyridine, α-, β- and γ-picoline, 2:6-, 2:4-, 2:5- and 2:3-lutidine, and 2:4:6-collidine. The precision is within ±2% and the preparation of the pure bases has been effected on larger columns. E. J. H. BIRCH

149. **Methods of analysis of the higher-boiling fractions of petroleum.** A. Crozier (Soc. Lubrizol, France). *Rev. Inst. Franç. Pétrole*, 1956, **11** (7–8), 869–912; (9), 1129–1160; (10), 1232–1268.—The constitution of the 200° to 300° fraction is discussed and the various methods, both physical and chemical, that are used to analyse such a fraction are reviewed. E. J. H. BIRCH

150. **Analysis of hydrocarbon blends by gas-liquid partition chromatography.** L. J. Nunez, W. H. Armstrong and H. W. Cogswell (Cities Service Res. and Development Co., Lake Charles, La., U.S.A.). *Anal. Chem.*, 1957, **29** (8), 1164–1165.—Analysis of nine hydrocarbon blends of known composition, on a commercially available vapour-phase chromatographic instrument, showed that the result for each component more closely approximated wt.-% than mole-%. The result for each component was calculated from the ratio of its peak area to the total of all peaks observed, and was then compared with the known composition of the blend expressed both as wt.-% and mole-%. Data for nine hydrocarbon blends are listed and show that, with results calculated as mole-%, total individual discrepancies may reach 20 mole-%, whereas the max. deviation encountered by wt.-% calculation was 4 wt.-%. G. P. COOK

151. **Rapid method of determining iron in transmission oils.** P. G. Filatov. *Zavod. Lab.*, 1957, **23** (6), 704–706.—The sample of oil is placed inside an induction coil in an a.c. circuit and measurements based on inductance, which varies only with the content of Fe in the oil, are carried out. The method, which takes only 3 or 4 min., is accurate to within ±0·008% of Fe. G. S. SMITH

152. **Measurement of the composition of engine oils during use.** G. V. Vinogradov, L. Ya. Semechkin and N. T. Pavlovskaya. *Zhur. Prikl. Khim.*, 1957, **30** (4), 657–660.—The oil dissolved in *iso*-octane is passed through a column of silica gel (250 cm high × 2·5 cm in diam.), which is then washed with *iso*octane until the pure solvent is coming through. The column is then washed with benzene until the pure solvent appears, then with ethanol - benzene (1 + 1) and finally with acetone to obtain resinous materials. The characteristics of the various fractions are determined. G. S. SMITH

153. **Analysis of volatile aerosol constituents. I.** M. J. Root and M. J. Maury (G. Barr & Co., Chicago). *Soap, N.Y.*, 1957, **33** (3), 75–78, 109; 1957, **33** (4), 101, 103, 105, 107.—The application of gas chromatography to the analysis of aerosols is reviewed and illustrated with reference to Freon mixtures. S.C.I. ABSTR.

154. **The application of physical methods of analysis in the gas industry.** A. B. Densham and G. Gough. *Gas World*, 1957, **146** (3), 805.—A review.

155. **Summative analysis of several Victorian brown coals.** P. Casamento and O. G. Ingles (C.S.I.R.O., Melbourne, Australia). *Aust. J. Appl. Sci.*, 1957, **8** (1), 55–61.—Analyses of 12 brown coals (H_2O = 55 to 65%) by the procedures of B.S. 1016:1942 show that the content of O as determined by difference can often be in error by 0·5 to 2·5%. Accurate and reproducible results for O can be obtained only by Spooner's method (*cf. Brit. Abstr. C*, 1947, 59) with a dry sample and modifications in the absorption train and packings. A total analysis of such coals (summation to 100%) can be calculated from ≃8 separate determinations (C, H, N, O, Cl, S, ash, and SO_3 in ash); this enables an undetermined or "suspect" component to be estimated to within 0·5%. W. J. BAKER

156. **Relative rank of Victorian brown coals.** O. G. Ingles (C.S.I.R.O., Melbourne, Australia). *Aust. J. Appl. Sci.*, 1957, **8** (1), 62–70.—Because of high contents of H_2O and anomalous contents of N and S, the usual assessment of coal rank from chemical analyses cannot be applied confidently to "earthy" or "lignitic" brown coals. Modifications and corrections for converting the analyses to a "pure coal" basis are given. Analyses should be reported on the dry, ash-free basis, and the calorific value should not be corrected for S. A method of calculating the combined H_2O in the original mineral matter of brown coal is given. Decrease in the content of O indicates rise in relative rank. W. J. BAKER

157. **Determination of ash in tar and pitch with low ash contents.** L. Wnękowska and J. Leśniak (Dept. Chem. of Coal, Mining Inst., Katowice). *Chem. Anal., Warsaw*, 1957, **2** (1), 29–34.—The usual methods for the determination of ash in organic samples by combustion in open crucibles were not considered sensitive or accurate enough at a concn. of ash of 0·03% or less. For samples containing 0·003 to 0·1% of ash, a method which consists in combustion of the sample (0·5 g) in a stream of O in a quartz combustion tube (18 mm × 100 cm) at 800° for about 6 hr. gives satisfactory results. K. F. SPOREK

158. **Paper chromatography of acid triphenylmethane dyes.** J. Dobáš (Res. Inst. Org. Synth., Org.-Technol. Lab., Pardubice-Rybitví, Czechoslovakia). *Chem. Listy*, 1957, **51** (6), 1202–1203.—With the use of the system butanol - pyridine - water as solvent, the separation of various acid triphenylmethane dyes (5 to 8 μg) is possible. J. ZÝKA

159. **Application of reverse-phase chromatography to the analysis of fat-soluble dyes.** M. R. Verma and Ramji Dass (Nat. Phys. Lab., New Delhi). *Naturwissenschaften*, 1957, **44** (12), 351 (in English).—The identity of commercial dyes can be established by ascending chromatography on Whatman No. 1 paper which has been either acetylated, or treated with Dow-Corning silicone 1107, liquid paraffin or oleic acid. The developing solvents are various aq. mixtures of organic solvents. E. KAWERAU

160. **Determination of very small quantities of mercury in synthetic dyes for colouring food.** H. Onrust (Keuringsdienst van Waren, Rotterdam,

Holland). *Chem. Weekbl.*, 1957, **53** (28), 383–386.—By the method described, 0·5 μg of Hg per gram of synthetic dye can be determined. The dye (5 g) is digested, in an all-glass apparatus, first with H_2SO_4 - HNO_3 and then with HNO_3 (sp. gr. 1·4) - $HClO_4$ (sp. gr. 1·67) (1 : 1) to obtain a clear colourless digest. The Hg is extracted from a fairly acid soln. with a $CHCl_3$ soln. of dithizone, then transferred to an aq. medium by destroying the dithizone complex with $KMnO_4$ in acid soln., and determined visually with dithizone by the mixed colour method of Belz and Reith (Draft Method 1956 for the Commission for Determination of Trace Elements in Food, I.U.P.A.C.). Several dyes have been analysed by this method and added amounts of Hg have been completely recovered within the limits of accuracy of the method. Triphenylmethane dyes tend to produce coloured and incompletely destroyed digests unless subjected to the HNO_3 - $HClO_4$ treatment described above. Erythrosine (a dye containing about 50% of iodine) cannot be analysed by this method owing to reduction of some iodine to HI, which combines with Hg to form a compound which resists extraction with dithizone. P. Haas

161. **Determination of 8-hydroxyquinoline (oxine) in mechanical pulp.** P. O. Bethge, O. T. Carlson and S. K. L. Freyschuss (Swedish Forest Products Res. Lab., Stockholm). *Svensk Papp.-Tidn.*, 1956, **59**, 591–592 (in English).—Direct extraction as the copper complex after acid digestion is suggested as a method for the determination of oxine (used as a fungicide) in mechanical pulp. A recovery of 90 to 100% has been attained. *Procedure*—Make three successive digestions of the cut-up air-dried sample (3 g) with 0·1 *M* HCl (35 ml), by heating under reflux for 1 hr. at 90°. Filter the combined extracts, cool and shake with $CHCl_3$ (20 ml). Filter again through a wet paper and wash out the flask with a further quantity of acid (5 ml). To the combined filtrates add 0·001 *M* $CuSO_4$ (2 ml) and 3 *M* Na acetate (15 ml), shake the mixture with $CHCl_3$ (20 ml) and run off the $CHCl_3$ layer through a plug of cotton wool into a 5-cm spectrophotometer cell. Determine the extinction of the soln. at 410 mμ. Prepare a calibration curve from $CHCl_3$ extracts of a mixture of 0·1 *M* HCl (100 ml) with measured quantities of standard oxine soln. To eliminate interference by resins in the sample, run a blank experiment in which the soln. of Cu is replaced by 2 ml of 0·05 *M* EDTA (disodium salt). D. G. Forbes

162. **Cerimetric determination of Heliogen blue SBL.** M. Matrka and F. Navrátil (Res. Inst. Org. Synth., Pardubice-Rybitví, Czechoslovakia). *Chem. Průmysl*, 1957, **7** (1), 21–22.—The method is based on the oxidation of the phthalocyanine mol. by ceric sulphate. *Procedure*—Dissolve the sample (0·5 to 1·0 g) in water (50 ml) and titrate potentiometrically with 0·1 *N* $Ce(SO_4)_2$. J. Böswart

163. **Determination of mercury in paper-mill white-water.** O. T. Carlson and B. Alderin (Swedish Forest Products Res. Lab., Stockholm). *Svensk Papp.-Tidn.*, 1956, **59**, 271–273 (in English).—An improved method, which gives an 85 to 100% yield, is described for the determination of Hg present as phenylmercury compounds in paper-mill white-water. A new apparatus is described. *Procedure*—Distil the sample (containing 10 to 50 μg of Hg) until most of the water is distilled. Cool the contents of the flask and add a mixture (1 : 1) of conc. H_2SO_4 and $HClO_4$ (sp. gr. 1·67) (10 ml for every 50 ml of the sample). Resume the distillation until the liquid in the flask is colourless, then for a further 3 min. Run the distillate into a beaker, washing down the condenser with water (3 × 25 ml) and adding this to the beaker. Transfer the reaction mixture from the flask to the beaker and wash it also with water (3 × 10 ml). Adjust the pH of the mixture to 1·0 with aq. NH_3 soln. (equal vol. of water and conc. aq. NH_3 soln.) (≃ 30 ml). Add a soln. of hydroxylamine sulphate (20%, previously shaken with a 0·001% soln. of dithizone in $CHCl_3$ until interfering metals are removed) (10 ml). Set the mixture aside overnight, then extract with dithizone soln. (20 ml). Filter a few ml of the $CHCl_3$ layer into a spectrophotometer cell through a cotton-wool plug and shake the remaining $CHCl_3$ with 20 ml of a soln. of K H phthalate (10·2 g) and KI (30 g) in 500 ml of water and filter the $CHCl_3$ layer as before into another photometer cell. Compare the extinctions of the $CHCl_3$ soln. at 620 mμ against pure $CHCl_3$ as a reference. Perform a blank determination and use a calibration curve prepared from a soln. obtained by extracting a mixture of water (150 ml), conc. acids (10 ml) and measured amounts of standard $HgCl_2$ soln. (10 to 50 μg of Hg). D. G. Forbes

164. **Determination of micro quantities of pentachlorophenol in pulp and paper.** G. Luedemann, I. Sobolev, R. Russell and E. Reed. *TAPPI*, 1957, **40**, 104–106.—Steam-distillation to separate the pentachlorophenol from the other components of the sample is followed by extraction of the distillate with $CHCl_3$, and colorimetric determination of the pentachlorophenol with methylene blue. The precision is about ±4%. A qual. and semi-quant. method, in which paper chromatography is used with β-methylumbelliferone as detecting agent, is also given. S. V. Sergeant

165. **Gravimetric and absorptiometric determination of lignin in pulp and wood.** V. Lorås and F. Løschbrandt. *Norsk Skogind.*, 1956, **10** (11), 402–413.—The suitability of two gravimetric methods, *i.e.*, a modified Halse method, with the use of a mixture of HCl and H_2SO_4, and the Swedish CCA5 standard method with 72% H_2SO_4, and of two absorptiometric methods, *i.e.*, the H_3PO_4 method developed by Bethge *et al.* and a method with 76% H_2SO_4, for the determination of lignin in sulphite and sulphate pulps has been investigated and the results are discussed. K. Wendtner

166. **Complexometric estimation of iron in cellulose.** B. Philipp and H. Hoyme (Inst. Textile Tech., Teltow-Seehof, Berlin). *Faserforsch. u. Textiltech.*, 1957, **8** (1), 34–35.—Full descriptions are given of the estimation of Fe in cellulose and in the caustic lyes used for steeping cellulose in viscose manufacture, or expressed from the alkali cellulose in such manufacture, by titration with EDTA with variamine blue as indicator. The method is more rapid and gives more reproducible results than does the colorimetric method; the results are usually within 1·9% of those of gravimetric determinations (as against 3·5% for the colorimetric method). There is no interference from Al, Mn, Cl^- and NO_3^-, but Cu can give a redox reaction with the indicator, with resulting high values for Fe; any Cu present should be < ≃ 3% of the Fe present. H. L. Whitehead

167. **Determination of sulphate acid ester in unstabilised cellulose nitrate.** P. E. Gagnon, K. F. Keirstead and D. Yamasaki (Laval Univ., Quebec, Canada). *Canad. J. Technol.*, 1957, **34** (8), 477–484. —Four methods analogous to those described previously (Keirstad and Myers, *Ibid.*, **34**, 71; *Canad. J. Chem.*, 1954, **32**, 566) are given, and their validity is assessed by reference to the results obtained by the gravimetric ($BaSO_4$) method. Satisfactory values for the content of sulphate ester in $\simeq 6$ g of moist sample (50% of H_2O) of unstabilised cellulose nitrate can be obtained by (*i*) direct titration, with 0·01 *N* NaOH, of a well-stirred mixture of the sample in H_2O, completing the titration within 10 to 20 min., (*ii*) titration with 0·01 *N* KOH after hydrolysis in acetone - H_2O at 25°, (*iii*) determining the content of Ca after ion exchange with 0·02 *N* $CaCl_2$, or (*iv*) determination of SO_4^{2-} after treatment with 50% ethanol.

W. J. Baker

168. **Quantitative estimation of a non-ionic detergent.** J. C. Griffith (School of Appl. Chem., N.S.W. Univ. of Technol., Sydney, Australia). *Chem. & Ind.*, 1957, (30), 1041–1042.—A u.v. spectrophotometric method is described for determining an ethylene oxide adduct (Triton X-100). There is a linear relationship between the concn. of dil. soln. and the optical density of the peak at 278 mμ (due to the aromatic ring) in the u.v. spectrum. The concn. of an unknown soln. can be determined to within 1% by reference to a standard curve.

A. M. Spratt

169. **Quantitative estimation of linalol in coriander oil.** H. Steindelówna. *Biul. Inst. Roślin Leczniczych*, 1957, **3** (2), 139–143.—An easy and practical method is given for the estimation of the essential oil by dehydration. A specially prepared mixture of Cu and CuO is used as catalyst, and the estimation is carried out in tetrachloroethane solution in the Deryng apparatus (*cf.* Polish Pharmacopoeia III). Results obtained differ by 1·26% from the theoretical value. Interference by geraniol is negligible.

H. Dmowska

170. **Investigation of the derivatives of caprolactam. I. Colour reactions of caprolactam.** K. Czerepko and H. Sikorska-Tomicka (Dept. of General Chem., Med. Acad., Białystok, Poland). *Chem. Anal., Warsaw*, 1957, **2** (1), 44–51.—Caprolactam forms characteristic ppt. with bismuth potassium iodide, mercuric potassium iodide and with soln. of iodine in KI. By means of these reactions caprolactam can be detected in soln. at concn. of a few micrograms per ml.

III. Detection of caprolactam in a polymer. K. Czerepko and L. Jaroszewicz. *Ibid.*, 1957, **2** (2), 173–175.—Two methods for the detection of caprolactam monomers in an aq. extract of a polymer are described. The reaction with bismuth potassium iodide in acid solution containing excess of KI gives an orange to red ppt. of characteristic cryst. structure. The colour of the ppt. depends on the acidity of the solution and on the proportion of the reagents. Less than 0·3% of caprolactam can be detected. Acid hydrolysis of caprolactam and reaction with ninhydrin in a solution neutralised with NaOH to a pH of 6·3 to 6·5 gives a violet - blue coloration, insoluble in ether and carbon tetrachloride, but gives a characteristic colour in chloroform and benzene. The colour of the solution depends on the pH and changes from cherry red to violet - blue, then to grey. With higher concn. of caprolactam, a brown crystalline compound is formed. Addition of acetone or alcohol stabilises the colour of the solution. Both methods can be used for quant. analysis.

K. F. Sporek
H. Dmowska

171. **Determination of the acid value of linseed oil extracted from paint films.** L. J. David. *Chim. Anal.*, 1957, **39** (6), 224–225.—The sample, after mechanical treatment to render it homogeneous, is mixed with ether. Pigment is removed by centrifuging, and the ether is distilled off at 50° to 60° in the presence of an inert gas; subsequent distillation under vacuum at 150° removes solvents. The distillate is mixed with ethanol - benzene, and a soln. of 0·1 *N* oxalic acid in ethanol is added. After heating for 1 hr. under reflux, the soln. is titrated with 0·1 *N* ethanolic KOH, with phenolphthalein as indicator. The acid value is calculated from a given formula.

W. J. Wright

172. **Infra-red spectroscopic analysis of varnish films on wire.** D. Hummel (Fa. Robert Bosch G.m.b.H., Stuttgart). *Farbe u. Lack*, 1956, **62**, 529–539.—The sample is best examined by the potassium bromide pellet technique, but if several coats are present the interpretation of the combined spectra may be difficult and some separation by treatment with selective solvents may be necessary. In some cases examination of the distillate and residue from a pyrolysed sample may be helpful. Chemical tests for elements (N, Cl, Si, P, S, etc.) should be carried out. Reference spectra of most of the types of varnish likely to be encountered are shown.

L. A. O'Neill

See also Abstracts 176, 220, 230, 315.

4.—BIOCHEMISTRY
INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE

Blood, Bile, Urine, etc.

173. **A method of measuring the pH of body fluids.** V. Wynn and J. Ludbrook (St. Mary's Hosp., London). *Lancet*, 1957, **i** (21), 1068–1071.—A constant-temp. pH cell, that enables measurement to be made on 0·5 ml of fluid under anaerobic conditions, is described. The measuring apparatus consists of a direct-reading millivoltmeter used in conjunction with a simple potentiometer. Readings are reproducible to 0·005 pH unit.

H. F. W. Kirkpatrick

174. **Quantitative determination of fibrinogen in blood by paper electrophoresis.** N. I. Lazarov. *Lab. Delo*, 1957, (4), 11–13.—The method is based on the fact that the γ-globulin fraction moves together with the fibrinogen of the plasma in the electric field. Fibrinogen can therefore be estimated from the difference in intensity of the spots obtained by electrophoresis of the plasma and the serum.

L. Herdan

175. **Micro procedure for the diphenylamine reaction with the blood of diseased persons.** E. G. Larskiĭ (Inst. Pediatr. AMH, Moscow). *Lab. Delo*, 1957, (4), 13–14.—For the reagent, a mixture of glacial acetic acid (90 ml) and conc. H_2SO_4 (10 ml) is used in which is dissolved diphenylamine (1 g), purified by recrystallisation from ether. For the

controls the same proportions of the acids are used without the addition of diphenylamine. A simplified procedure is described in detail. L. HERDAN

176. **Carbon determination in biological material with a persulphate oxidation method.** S. L. Chen and K. J. H. Lauer (Red Star Yeast and Products Co., Milwaukee, Wis., U.S.A.). *Anal. Chem.*, 1957, **29** (8), 1225–1226.—The method of Katz *et al.* (*Anal. Abstr.*, 1955, **2**, 516) has been applied to biological materials and some water-insol. organic compounds. A modification in the preparation of the samples involved dissolving the sample in conc. H_2SO_4 before its addition to the combustion-diffusion vessel. Recoveries of C from various samples were >96%. G. P. COOK

177. **Carbon determination with alkaline persulphate in the Warburg manometer.** E. H. Battley (Hopkins Marine Sta., Stanford Univ., Pacific Grove, Calif., U.S.A.). *J. Biol. Chem.*, 1957, **226** (1), 237–240.—A convenient manometric method is described which requires less than 250 μg of C. Two determinations can be made simultaneously in triplicate. The results show that the method is sufficiently accurate for most determinations required in biological work. It is useful in checking the extent to which C remains as unidentified compounds in a culture medium at termination of growth. J. N. ASHLEY

178. **Separation and determination of microgram quantities of tellurium in urine.** C. K. Hanson (Univ. Utah, Salt Lake City, U.S.A.). *Anal. Chem.*, 1957, **29** (8), 1204–1206.—Microgram amounts of Te can be separated from all but a few elements by extraction into *n*-pentanol from a soln. approx. *N* in H^+ and 0·6 *N* in I^-. The extract is washed with *N* HI and the solvent is removed by evaporation. The residue is redissolved in HCl from which the Te is pptd. in finely divided form by the addition of $SnCl_2$. After mixing, the soln. is transferred to a colorimeter where the transmittance is measured at 440 mμ. Recoveries of Te from standard samples were >94·5%. On application to urine samples, a mean recovery of 100·3% with a standard deviation of ±0·8% was obtained from 44 samples containing 10 μg of added Te, and recoveries and precision at the 20-μg level were similar. Interference is caused by Hg, Au, Pd, Pt, Rh and Ir. G. P. COOK

179. **Comparison between three methods used for the preparation of tissues for determinations of potassium and sodium.** M. S. Mounib and J. V. Evans (Rowett Res. Inst., Bucksburn, Aberdeenshire, Gt. Britain). *Analyst*, 1957, **52**, 522–523.—The three methods compared are (*a*) digestion with trichloroacetic acid, (*b*) boiling with water under reflux, and (*c*) boiling as in (*b*) with the subsequent addition of 1 drop of HNO_3 before storage of the digest in the refrigerator. Potassium and sodium were determined by flame photometer. With the heart, liver, kidney and spleen (of sheep), method (*a*) gave higher results for K than method (*b*), but with skeletal tissue there was no significant difference with these two methods. Methods (*a*) and (*c*) gave similar values for K in all tissues except the spleen; with this, method (*a*) gave higher concn. than method (*c*). Significant differences in concn. of Na as determined by the three methods occurred only with the kidney and spleen. Acidification, unnecessary for complete liberation of K from skeletal muscle, is necessary with heart, liver, kidney and spleen. This confirms the suggestion made by numerous workers that K may exist in more than one form in a tissue, and that its state of combination varies from one organ to another. A. O. JONES

180. **Studies in histochemistry. XLVII. Microdetermination of magnesium and its histological distribution in the adrenal in various functional states.** D. Glick, E. F. Freier and M. J. Ochs (Dept. of Physiol. Chem., Univ. of Minnesota Med. Sch., Minneapolis, U.S.A.). *J. Biol. Chem.*, 1957, **226** (1), 77–82.—A photometric micromethod, based on a modification of the Titan yellow method, is described for the determination of Mg in microgram samples of tissue. It depends on the colour change from yellow to red which the dye undergoes when $Mg(OH)_2$ is pptd. in its presence. J. N. ASHLEY

181. **A method of assaying small amounts of sulphur-35 in tissues.** C.-H. Dohlman (Chem. Dept. II, Karolinska Inst., Stockholm). *Ark. Kem.*, 1957, **11** (3), 255–257 (in English).—Tissue specimens (≯60 mg. dry weight) are treated with 0·2 ml of 2% H_2SO_4 and 2 ml of fuming HNO_3, and heated under reflux for 0·5 hr. at 80° in 10-ml test-tubes. The tubes are then heated to between 110° and 120° without condensers and evaporated to dryness, and the cooled residue is heated with 2 ml of 30% H_2O_2 for 0·5 hr. at 90°, and then evaporated to dryness at 100° to 110°. The ash is dissolved in 2 ml of water and evaporated on lead planchets. The count rate is determined under a thin-window Geiger-Müller tube. The standard deviation of the determinations is ±2·1%. The recovery is lowered if the dry weight of tissue exceeds 60 mg. E. J. H. BIRCH

182. **Cellular constituents. Major and minor metals in normal and abnormal tissues. I. Analysis of Wistar rat livers for copper, iron, magnesium, manganese, molybdenum and zinc.** F. Bergel, J. L. Everett, J. B. Martin and J. S. Webb (Chester Beatty Res. Inst., Royal Cancer Hospital, London). *J. Pharm. Pharmacol.*, 1957, **9** (8), 522–531.—Emission spectroscopy has been used for comparative analyses for these metals in rat liver. The biochemical significance of the results is discussed. *Procedure*—Dry the sample at 110° for 4 hr., powder and dry at 110° to constant wt. De-fat by Soxhlet extraction with ether. Incinerate in a muffle-furnace at 435° for 7 hr., cool, grind and mix with NH_4NO_3 (few crystals) and heat at 435° for a further 24 hr. Working in quadruplicate, mix an aliquot (≃20 mg) of the ash with pure C powder and compress into the bore of a Jelfke carbon electrode. Obtain the spectrogram from 270 to 480 mμ by burning the sample to completion by the cathode layer arc technique. Read the concn. graphically from a working curve which relates log concn. and log percentage relative density of the spectral line. The coeff. of variation of results, based on four samples in each case, is of the order of 10%. A. R. ROGERS

183. **Determination of chloral hydrate and its transformation products in body fluids and tissues.** A. E. Meyer and R. Per Lee-Motter (Fellows Pharm. Lab., New York). *Arzneimittel-Forsch.*, 1957, **7** (3), 194–197.—A method is described for determining chloral hydrate (**I**), trichloroacetic acid (**II**), trichloroethanol (**III**) and urochloralic acid (**IV**) in body fluids (blood, milk, urine, etc.), which is based

on a Fujiwara colorimetric determination of **II** (or of **I** plus **II**; each give the same line) after treatment with conc. aq. NaOH and pyridine. *Procedure*—The sample fluid, after suitable dilution (or concn. for urine) is divided into four measured fractions; protein-containing fluids must first be freed from protein by treatment with sodium tungstate and 1/12 *N* H_2SO_4. Fraction *A*, for the estimation of **I** and **II** together, is added to pyridine, followed by the alkali (this order is necessary to avoid subsequent decomp. of **I**). The mixture is heated for 2 min. at water-bath temp., then cooled in ice, and the colorimetric determination is made (at concn. of 1 to 40 μg per ml) photometrically or with an electric colorimeter and a 520 filter. **II** (and hence **I** by difference) is similarly determined in fraction *B*, but the fraction is first mixed with the alkali and set aside for 1 hr. to decompose **I** before adding pyridine and continuing the procedure as described above. Fraction *C* is rapidly evaporated at 100° (rapidity is essential) to remove **I** and **III**; the residue is treated with Cr_2O_3 and conc. H_2SO_4 to oxidise **IV** to **II**; then total **II** (initial **II** and that from **IV**) is determined as described above after adding pyridine and alkali, etc. The figure for **IV** is calculated by using the figure for initial **II** obtained with fraction *B*. Fraction *D* is treated similarly to *C*, but the evaporation step is omitted; all four compounds are thus in the form of **II** and are so estimated. The figure for **III** is calculated by the use of the values determined for **I**, **II** and **IV**. H. L. WHITEHEAD

184. **Rapid colorimetric method for the determination of *iso*nicotinic acid hydrazide (isoniazid) in blood serum.** V. Scardi (Inst. of Human Physiol., Univ. of Naples, Italy). *Clin. Chim. Acta*, 1957, **2** (2), 134–139 (in English).—Add serum or plasma (1 ml) to H_2O (2 ml) in a centrifuge tube, mix well, add 0·05 *N* acetic acid (1 ml) and mix. Heat the tube in a boiling-water bath for 2 to 3 min. and centrifuge for 10 min. at 3000 r.p.m. To the clear supernatant fluid (2 ml) add buffer soln. [0·2 *M* $Na_2HPO_4.2H_2O$ (14 ml) and 0·1 *M* citric acid (6 ml)] (1·5 ml) and 0·2% Na pentacyanoamminoferroate in 0·02 *N* aq. NH_3 (0·5 ml). Mix and, after 10 min., measure the absorption at 430 mμ. To standardise, use 2 ml of aq. soln. of isoniazid containing 0 to 100 μg of the pure substance for the colour test; a blank is run with the test. To prepare the Na pentacyanoamminoferroate, grind sodium nitroprusside (10 g) to a fine powder and treat with conc. aq. NH_3 (35 ml) overnight in a refrigerator. Wash the yellow product repeatedly with ethanol and finally with ether. Store the dry substance in a desiccator. H. F. W. KIRKPATRICK

185. **A very simple fluorescent method of determining acrichine [mepacrine] and stilbamidine in urine.** V. G. Mikhaĭlov (Tashkent Med. Inst.). *Lab. Delo*, 1957, (4), 15–16.—A drop of urine, dried on filter-paper, leaves a residue which fluoresces pale blue. If stilbamidine be present, a bright blue ring appears on the filter-paper in u.v. light, the intensity of this fluorescence depending on the concn. of the drug in the urine. If mepacrine be present in the urine, a yellow-greenish ring appears. The amount of drug can be estimated by means of colour standards which are stable for 2 to 3 months. L. HERDAN

186. **Colorimetric measurement of uric acid.** R. M. Archibald (Rockefeller Inst. Med. Res., New York, U.S.A.). *Clin. Chem.*, 1957, **3** (2), 102–105.—The method of Kern and Strausky (*Biochem. Z.*, 1937, **290**, 419) is modified to prevent the formation of turbidity and the colour reagent is used for protein pptn. H. F. W. KIRKPATRICK

187. **Effect of various protein precipitants on recoveries of creatinine added to plasma.** J. F. Van Pilsum and M. Bovis (Dept. of Physiol. Chem., Univ. of Minnesota, Minneapolis, U.S.A.). *Clin. Chem.*, 1957, **3** (2), 90–94.—Complete recovery of creatinine is obtained with tungstic acid, trichloroacetic acid, perchloric acid and cadmium hydroxide. Barium hydroxide with $ZnSO_4$ and NaOH with $ZnSO_4$ give incomplete recoveries. H. F. W. KIRKPATRICK

188. **Separation of adrenaline, noradrenaline and hydroxytyramine by ion-exchange chromatography.** N. Kirshner and M. Goodall (Dept. of Physiol., Duke Univ. Sch. of Med., Durham, N. Carolina, U.S.A.). *J. Biol. Chem.*, 1957, **226** (1), 207–212.—Amberlite IRC-50 is used for the separation. The recoveries from pure soln. are adrenaline 85 to 95%, noradrenaline 95 to 97%, and hydroxytyramine 83 to 95%. With trichloroacetic acid extracts of adrenal glands the recoveries are adrenaline 88 to 103% and noradrenaline 73 to 103%. The separation needs less time than that required by other methods, and is applicable to 0·02 to 1·0 mg of mixture. J. N. ASHLEY

189. **Polarography of 3:4-dihydroxy-α-methylaminoacetophenone (adrenalone).** G. Dušinský (State Inst. for Control of Drugs, Bratislava, Czechoslovakia). *Československ. Farm.*, 1956, **5** (4), 196–199.—Adrenalone (**I**) is reducible at the dropping mercury electrode in two waves in the whole range of pH. For quant. determination, 0·025 *M* $Na_2B_4O_7$ was used as supporting electrolyte, in which medium the oxidative degradation of **I** can also be investigated. After oxidation, two further, more positive waves appear. Soln. containing 5 to 40 mg % of **I** were analysed, and the wave with $E_{\frac{1}{2}} = -1{\cdot}5$ V was measured for evaluation (calibration curve). Adrenaline and other phenolic compounds do not interfere, and the method is more selective than colorimetry or titrimetry. J. VOLKE

190. **Application of various methods to quantitative estimation of 5-hydroxytryptamine and 5-hydroxyindolylacetic acid in a case of metastasising intestinal carcinoids.** W. Zylka and T. Köhler (Med. Poliklinik, Univ. Köln, Germany). *Klin. Wochschr.*, 1957, **35** (12), 622–626.—An investigation of the body fluids and tumour tissue by means of biological tests, high-voltage electrophoresis, paper chromatography, colorimetric and spectrophotometric analysis is described. H. F. W. KIRKPATRICK

191. **Determination of hexosamines in conjunction with electrophoresis on starch.** O. W. Neuhaus and M. Letzring (Wayne State Univ. Coll. of Med., Detroit, Mich., U.S.A.). *Anal. Chem.*, 1957, **29** (8), 1230–1233.—Hexosamines in the eluates from electrophoretic separations on starch must be separated from interfering substances before determination with Ehrlich reagent. This is achieved by the extraction, with *iso*amyl alcohol, of the compounds formed by hexosamines and acetylacetone. A typical glycoprotein curve obtained by the electrophoretic separation of human serum is also presented. The extraction procedure may also be used to determine hexosamines in 0·01 to 0·02 ml of serum. Recoveries of 90% were obtained for all levels of glucosamine in the presence of 2 mg of protein. G. P. COOK

192. **Some observations on the measurement of "binding" of cyanocobalamin by "intrinsic factor" preparations.** M. E. Gregory and E. S. Holdsworth (Nat. Inst. for Res. in Dairying, Shinfield, Berks., England). *Biochem. J.*, 1957, **66** (3), 456–462.—Cyanocobalamin bound to extracts of pig pyloric mucosa (intrinsic factor concentrates) is active for *Escherichia coli* in plate assay techniques and hence the use of plate assays, and possibly other assays, with micro-organisms for the determination of binding activity is unreliable. The previously described ultrafiltration method (*Ibid.*, 1955, **59**, 329) for measuring binding capacity is extended by the use of radioactive cyanocobalamin. The method is reliable and its advantages over the dialysis method are discussed. The technique is unaffected by lysozyme, heparin, chondroitinsulphate and nucleic acid, all of which interfere with methods that depend on the uptake of free vitamin by bacteria.
J. N. ASHLEY

193. **Determination of bound vitamin B_{12}.** O. N. Miller (Tulane Univ. Med. Sch., New Orleans, La., U.S.A.). *Arch. Biochem. Biophys.*, 1957, **68** (2), 255–262.—Free vitamin B_{12} is removed from serum or plasma by shaking mechanically with activated charcoal (75 mg per ml of sample) for 15 min. The suspension is centrifuged at 25,000 ***g*** for 15 min., at 5°, and the protein-bound vitamin-B_{12} content of the supernatant liquid is determined microbiologically. Total B_{12} activity is determined on a separate portion of the sample and free vitamin B_{12} is calculated by difference. In normal serum, 80 to 85% of the B_{12} activity is present in the bound form.
W. H. C. SHAW

194. **Enzymic formation of xylulose 5-phosphate from ribose 5-phosphate in spleen. [Determination of ribulose and xylulose 5-phosphates.]** G. Ashwell and J. Hickman (Nat. Inst. of Arthritis and Metabolic Diseases, Nat. Inst. of Health, Bethesda, Md., U.S.A.). *J. Biol. Chem.*, 1957, **226** (1), 65–76.—The simultaneous determination of these phosphates is described. Xylulose 5-phosphate is determined by the transketolase reaction in the triose phosphate dehydrogenase system described by Cori *et al.* (*Brit. Abstr. A III*, 1948, 1085). This is a rapid and specific method for the determination of this sugar phosphate either alone or in mixtures. The cysteine - carbazole reaction of Dische and Borenfreund (*Brit. Abstr. C*, 1952, 54) is used to determine the ribulose 5-phosphate. Since both ribulose and xylulose are determined by this method, the xylulose 5-phosphate is first determined by the enzyme assay described above, and the appropriate correction is made.
J. N. ASHLEY

195. **The micro-estimation, distribution and biosynthesis of 2:3-diphosphoglyceric acid.** J. C. Towne, V. W. Rodwell and S. Grisolia (Dept. of Med., Univ. of Kansas Med. Centre, Kansas City, U.S.A.). *J. Biol. Chem.*, 1957, **226** (2), 777–788.—Four methods are described for the determination of the acid. They all depend on the catalytic effect of the acid on phosphoglyceric acid mutase.
J. N. ASHLEY

196. **Studies on phospholipids. I. The determination of amino nitrogen in unhydrolysed phospholipids.** L. W. Wheeldon and F. D. Collins (Dept. of Biochem., John Curtin Sch. of Med. Res., Australian Nat. Univ., Canberra). *Biochem. J.*, 1957, **66** (3), 435–441.—1-Fluoro-2:4-dinitrobenzene reacts in the presence of triethylamine with the free amino groups of phospholipids dissolved in benzene. The extinction of the dinitrophenyllipids is measured spectrophotometrically at 345 mμ (ε 16,600) or 400 mμ (ε 6200) in light petroleum. The method is more reliable than the ninhydrin procedure, the reagent is more stable, the blank is negligible and there is no need for a standard. The method will determine 0·02 μmole, but the usual range is 0·1 to 2·5 μmoles. Lecithin and other lipids do not interfere, but special precautions are necessary when low concn. of kephalin are present.
J. N. ASHLEY

197. **Paper chromatography of phospholipids with solvent mixtures of ketones and acetic acid.** R. F. Wittner, G. V. Marinetti, A. Morrison and L. Heicklin (Rochester Univ., Med. Sch., New York, U.S.A.). *Arch. Biochem. Biophys.*, 1957, **68** (1), 15–20.—Separation of many phospholipids is achieved on acetic acid-washed Whatman No. 1 paper by the ascending method after equilibration of the paper for 24 hr. at 28°. Development is for 3 to 7 hr. at 28° with one of the following solvents, for which R_F values are listed—pentan-2-one - acetic acid (30:2), 3-methylbutan-2-one - acetic acid (30:3), 4-methylpentan-2-one - acetic acid (30:2) or 2:6-dimethylheptan-4-one - acetic acid (30:7). For satisfactory separation, 10 to 25 μg of phospholipid in 25 μl of soln. is required, and the compounds are detected with Rhodamine B or G, or, when appropriate, with ninhydrin or a test for choline.
W. H. C. SHAW

198. **Quantitative estimation of sialic acids [neuraminic acids]. I. A colorimetric method with orcinol - hydrochloric acid (Bial's) reagent.** L. Svennerholm (Dept. of Med. Biochem., Univ. of Gothenburg, Sweden). *Ark. Kemi*, 1957, **10** (6), 577–596 (in English).—An investigation of the optimal conditions for the quant. estimation of sialic acids with Bial's reagent has shown that Cu^{2+} may be used to replace Fe^{3+} without loss of efficiency. *Procedure*—Samples (2 ml, containing 10 to 40 μg of sialic acid) are pipetted into three glass-stoppered tubes. Orcinol reagent [0·2 g of orcinol in water (10 ml), conc. HCl (80 ml), 0·1 *M* Fe^{3+} (1 ml) or 0·1 *M* Cu^{2+} (0·25 ml), made up to 100 ml with water] (2 ml) is added to two of the tubes, and 2 ml of this reagent without the orcinol to the third, as blank. The tubes are heated at 106° for 15 min., cooled to room temp. and *iso*amyl alcohol (5 ml) is added. The tubes are shaken vigorously, cooled in ice water for 10 min., and centrifuged for 1 min. at 500 to 1000 r.p.m. The soln. are kept in iced water until the alcohol phase is separated for spectrophotometric measurement at 575 and 680 mμ. Readings should be made within 1 hr. after the heating.

II. A colorimetric resorcinol - hydrochloric acid method. L. Svennerholm. *Biochim. Biophys. Acta*, 1957, **24** (3), 604–611.—N-Acetyl-, N-glycollyl- and NO-diacetyl-sialic acids (10 to 30 μg) are determined with a resorcinol - HCl reagent containing $CuSO_4$. The colour formed by heating is extracted into amyl alcohol and the compounds are calc. by the methods given from the extinctions at 450 and 580 mμ and comparison with standards. The sensitivity is greater than that attainable with the orcinol - HCl method, and interference by carbohydrates is less. The determination of sialic acids in body fluids is discussed.
C. A. SLATER
W. H. C. SHAW

199. **Paper chromatography of 56 amino compounds using phenol and butanol - acetic acid as solvents.** T. E. Parry (Llandough Hosp., Penarth,

Glam., Wales). *Clin. Chim. Acta*, 1957, **2** (2), 115–125.—The offensive and persistent odour of collidine is a serious disadvantage to its routine use but lack of comprehensive data for other solvent systems has limited their use. The characteristics of 56 amino acids and related compounds on two-dimensional chromatograms when phenol, followed by *n*-butanol - acetic acid - H_2O, is used, are presented.

H. F. W. KIRKPATRICK

200. **Formol titration: an evaluation of its various modifications.** W. H. Taylor (Dept. of Clinical Biochem., Radcliffe Infirmary, Univ. of Oxford). *Analyst*, 1957, **82**, 488–498.—The principles underlying the formol titration method of determining amino-acid N under various experimental conditions are examined with a view to resolving the confusion surrounding the procedures used. Titrations were made potentiometrically and with indicators, and it was found that, under conditions simulating protein hydrolysis and with a final concn. of formaldehyde of 6 to 9%, a direct titration from the pH of hydrolysis to pH 8·5 gives reasonably quant. results. Indirect procedures, in which the pH is adjusted before addition of formaldehyde, do not yield reproducible results, and in the adjustment there may be titration of part of the amino acid or peptide. With preliminary neutralisation to pH 9, amino acids are under-estimated slightly and peptides severely. With preliminary neutralisation to pH 7, peptides are under-estimated slightly, but amino acids are not.

A. O. JONES

201. **The hydroxyproline content of animal tissue.** J. N. Aronson and C. A. Elvehjem (Coll. of Agric., Univ. of Wisconsin, Madison, U.S.A.). *Food Res.*, 1956, **21**, 109–111.—Hydroxyproline and proline have been detected in animal tissue, and account for a significant amount of the total nitrogen. A method is described for the determination of these compounds, which is based on the formation of a red coloration with ninhydrin. In simulated natural products, a recovery of 90% was obtained, and 98% when testing a mixture of the pure compounds. *Procedure*—Hydrolyse the fat-free sample by heating with 5 *N* KOH (25 ml per g of sample) in metal beakers at 121° and 15 lb pressure for 5 hr. Cool, set the beakers aside in a refrigerator overnight, then neutralise the contents with 10 *N* HCl. Make up to a convenient vol. with 0·1 *M* phosphate buffer. Into a 10-ml flask measure the sample (0·1 ml), phosphate buffer soln. (0·1 ml) and benzene (2·5 ml), and place the flask in a steam-heated water bath at 75°. Add a soln. of ninhydrin (0·3 *M* in 2-methoxyethanol) (0·2 ml), and shake the flask for 5 min., allowing minimum evaporation to occur. Remove the flask from the bath, dilute the soln. to vol. with benzene, and measure the benzene layer spectrophotometrically at 570 and 550 mμ for hydroxyproline and proline, respectively.

D. G. FORBES

202. **Determination of L-tyrosine as tyramine in urine.** R. E. Tashian (Inst. for Study of Human Variation, Columbia Univ., New York, U.S.A.). *Clin. Chem.*, 1957, **3** (2), 106–109.—Tyrosine is converted into tyramine by treatment of the urine with an acetone-dried powder prep. of *Streptococcus faecalis*. The tyramine is extracted into ethyl acetate, then into HCl, and determined by the nitrosonaphthol method.

H. F. W. KIRKPATRICK

203. **Colorimetric determination of tyrosine.** G. Ceriotti and L. Spandrio ("Centro Tumori" di Busto Arsizio, Italy). *Biochem. J.*, 1957, **66** (4), 607–610.—An improved method for the determination of tyrosine by the Gerngross *et al.* method (*Brit. Abstr. A*, 1933, 407) is described. The soln. of 1-nitroso-2-naphthol is prepared in 0·1 *N* NaOH instead of in ethanol; this stabilises the final colour and is an improvement on the method described by Thomas (*Brit. Abstr. C*, 1945, 133). The method is preferable to that of Udenfriend and Cooper (*Brit. Abstr. C*, 1953, 215) since it avoids the extraction of the excess of nitroso compound with 1:2-dichloroethane. The method is applicable to the determination of tyrosine in proteins, and appears to be specific except for tyramine, which is not usually present in protein hydrolysates.

J. N. ASHLEY

204. **Determination of tyrosine and tryptophan in proteins.** W. L. Bencze and K. Schmid (Harvard Med. Sch., Boston, Mass., U.S.A.). *Anal. Chem.*, 1957, **29** (8), 1193–1196.—The method is based on the measurement of absorbance in the range between 278 and 293 mμ. In order to diminish the error caused by the bathochromic shift in the absorption spectra of tyrosine and tryptophan, two new features are introduced. First, the slope of the line drawn tangentially to the two characteristic max. indicates the ratio of tyrosine to tryptophan, and second, the extinction at the higher max. is a measure of the tyrosine - tryptophan content.

G. P. COOK

205. **An improved method for the micro-determination of arginine by use of 8-hydroxyquinoline.** G. Ceriotti and L. Spandrio ("Centro Tumori" di Busto Arsizio, Italy). *Biochem. J.*, 1957, **66** (4), 603–607.—The colour reaction given by arginine with 8-hydroxyquinoline in the presence of NaOBr (Sakaguchi, *Brit. Abstr. C*, 1949, 402) is improved and the colour stabilised by extraction with butanol. The use of urea is avoided and the colour, which is stable for at least 12 hr., is measured spectrophotometrically at 507 mμ. The method determines accurately 0·5 to 6 μg of arginine per ml, and the results are const. and reproducible (standard deviation ±1·3%). The sensitivity of the original method is further increased because the vol. of the final coloured soln. is decreased. The method of Rosenberg *et al.* (*Anal. Abstr.*, 1956, **3**, 2822), which utilises a 1-naphthol - diacetyl reagent in the presence of propanol, is less sensitive and less reproducible. The modified method is applicable to the determination of arginine in protein hydrolysates.

J. N. ASHLEY

206. **Optical methods for measuring nucleoprotein and nucleic acid concentrations.** S. W. Englander and H. T. Epstein (Pittsburgh Univ., Pa., U.S.A.). *Arch. Biochem. Biophys.*, 1957, **68** (1), 144–149.—Nucleoprotein is estimated in virus preparations by means of a differential refractometer, and the concn. is calculated by the method given. Ribo- and deoxyribo-nucleic acids are determined from the extinction at 260 mμ at pH 5·7 and 7·0, respectively, after correction for absorption by protein and for scattering. Sensitivity and accuracy compare well with those attainable in standard biochemical methods.

W. H. C. SHAW

207. **A stable standard for the colorimetric determination of total protein, albumin, globulin and fibrinogen.** F. Rappaport and M. Loew (Beilinson Hosp., Petah Tikva, Israel). *Clin. Chim. Acta*, 1957, **2** (2), 126–130.—The prep. of 10% peptone soln. suitable for use as a standard for protein estimation by the biuret method is described.

H. F. W. KIRKPATRICK

208. **Further studies on the reaction of disulphides with silver nitrate. [Determination of disulphides.]** R. Cecil and J. R. McPhee (Dept. of Biochem., Univ. of Oxford). *Biochem. J.*, 1957, **66** (3), 538–543.—Conditions are given for the determination of the disulphides derived from glutathione—β-mercaptopropionic acid, homocysteine, cysteine N-formylcysteine, N-formylhomocysteine, β-aminoethanethiol and N-acetylcysteine ester. The disulphides are reduced by Na_2SO_3 to the thiols, which are then titrated potentiometrically with $HgCl_2$ after adjusting to a pH of 1·8 to 2 with dil. HNO_3. This $HgCl_2$ method is more convenient than the $AgNO_3$ method, which involves back-titration with KBr; moreover, Hg^{2+} are less liable than Ag^+ to form complexes with thiols.

J. N. ASHLEY

209. **Alpha- and beta-lipoprotein cholesterol. Method of rapid quantitative determination.** F. S. Nury and E. R. B. Smith (Walter Reed Army Inst. of Res., Washington, D.C., U.S.A.). *Clin. Chem.*, 1957, **3** (2), 110–115.—The method of Langan *et al.* (*J. Clin. Invest.*, 1955, **34**, 1427) is simplified and the time of analysis greatly reduced.

H. F. W. KIRKPATRICK

210. **Comparative value of X-ray diffraction and infra-red spectrophotometry in identifying certain sterols and their digitonides.** W. T. Beher, J. Parsons and G. D. Baker (Henry Ford Hospital, Detroit, Mich., U.S.A.). *Anal. Chem.*, 1957, **29** (8), 1147–1151.—Sterols in the C_{27} to C_{29} series can be easily differentiated by X-ray diffraction powder methods, but cannot be identified by i.r. spectrophotometry. Sterol digitonides cannot be identified by either method. X-ray diffraction establishes the digitonides as chemical compounds, whereas i.r. spectrophotometry does not distinguish between a simple mixture and a corresponding digitonide.

K. A. PROCTOR

211. **Determination of 17:21-dihydroxy-20-keto-steroids in blood plasma.** K. Eik-Nes (Dept. of Biol. Chem., Univ. of Utah Coll. of Med., Salt Lake City, U.S.A.). *J. Clin. Endocrin. & Metabolism*, 1957, **17** (4), 502–511.—For routine determinations by the Porter - Silber colour reaction, purification of the organic extract of plasma by a water - benzene partition step is preferred to chromatography on Florisil.

H. F. W. KIRKPATRICK

212. **Determination of oestrogen in urine.** H. Breuer, W. Nocke, G. Geissler and F. L. Mitchell (Chem. Abteilung, Chirurg. Univ.-Klinik, Bonn, Germany). *Klin. Wochschr.*, 1957, **35** (13), 672–674.—The method of Brown (*cf. Anal. Abstr.*, 1955, **2**, 3161) is shown to give reproducible results in agreement with those of biological tests. The specificity of the method is demonstrated by determinations of oestrogen excretion before and after oophorectomy.

H. F. W. KIRKPATRICK

213. **Determination of "pancreatitis lipase" in serum.** R. J. Henry, C. Sobel and S. Berkman (Bio-Science Lab., Los Angeles, Calif., U.S.A.). *Clin. Chem.*, 1957, **3** (2), 77–89.—The lipase occurring in the sera of patients with acute pancreatitis is specifically determined when olive oil is used as substrate. The method of Cherry and Crandall (*Amer. J. Physiol.*, 1932, **100**, 266) is recommended, with the substitution of thymolphthalein as indicator in the titration. Normal ranges are given as up to 0·9 unit for 4-hr. incubation and up to 1·5 units for 16 to 24-hr. incubation.

H. F. W. KIRKPATRICK

214. **Pyridoxamine phosphate transaminase. [Determination of enzyme activity.]** R. B. Beechey and F. C. Happold (Dept. of Biochem., Univ. Leeds, England). *Biochem. J.*, 1957, **66** (3), 520–527.—The enzyme, which causes the interconversion of pyridoxal phosphate and pyridoxamine phosphate, is determined spectrophotometrically. The absorption of light at 390 mμ by pyridoxal phosphate is max. at pH 14, whereas pyridoxamine phosphate does not absorb light at this wavelength and pH. If the transaminase reaction is stopped by addition of NaOH to the reaction mixture and the changes in light absorption at 390 mμ are measured, the rate of formation, or conversion, of pyridoxal phosphate is obtained. Transaminase activity is expressed as micrograms of pyridoxal phosphate produced from, or converted into, pyridoxamine phosphate.

J. N. ASHLEY

215. **Adenosine triphosphate - adenosine monophosphate transphosphorylase (myokinase). I. Isolation of the crystalline enzyme from rabbit skeletal muscle. [Determination of enzyme activity.]** L. Noda and S. A. Kuby (Inst. Enzyme Res., Univ. Wisconsin, Madison, U.S.A.). *J. Biol. Chem.*, 1957, **226** (1), 541–549.—A method is described for the determination of adenosine triphosphate - adenosine monophosphate transphosphorylase activity by the use of adenosine triphosphate - creatine transphosphorylase. This is effected by coupling the activities of the two enzymes in the presence of excess of creatine. The high-energy creatine phosphate so formed is determined as inorg. phosphate by King's method (*Brit. Abstr. A*, 1932, 786) after decomposition in acid - molybdate as described by Kuby *et al.* (*J. Biol. Chem.*, 1954, **209**, 191).

J. N. ASHLEY

See also Abstracts 286, 301, 312, 314, 335.

Drugs

216. **The use of paper chromatography in the control of mixtures of pharmaceuticals.** I. Jakubec, V. Lašková and E. Slámová (Inst. Pharm. Chem., Masaryk Univ., Brno, Czechoslovakia). *Farmácia*, 1956, **25** (5), 137–143.—Paper chromatography has been used successfully for the detection of acetylsalicylic acid, amidopyrine, phenazone, cinchophen, quinine salts, and, in some cases, of codeine in prepared mixtures (powders). Mixtures of water, low-boiling alcohol (methanol, ethanol or *iso*propyl alcohol) and aq. NH_3 have been found to be the best solvents, and a mixture of $FeCl_3$ with $K_3Fe(CN)_6$ the best detecting agent.

J. ZÝKA

217. **Radiometric methods in pharmaceutical analysis.** J. Tölgyessy, J. Majer and P. Schiller (Inst. Anal. Chem., Pharmac. Fac., Bratislava, Czechoslovakia). *Farmácia*, 1957, **26** (2), 41–47.—A survey of the principles of radiometric titrations is presented, and, as an example of this technique, the titration of mercuric salts with a soln. of KI is described.

J. ZÝKA

218. **Titrations of acids in anhydrous pyridine.** J. Vacek and J. Kráčmar (State Inst. for Control of Drugs, Prague, Czechoslovakia). *Českosl. Farm.*, 1956, **5** (2), 80–84.—The following substances may be titrated in anhydrous pyridine with Na methoxide as reagent, and phenolphthalein (in methanol) as indicator—(*a*) salicylic, acetylsalicylic, *p*-aminobenzoic, *p*-aminosalicylic, barbituric, *iso*nicotinic,

nicotinic, phenylcinchoninic, picric and sulphanilic acids (average error ±0·3%), and *p*-benzamidosalicylic acid (average error ±5%); (*b*) allobarbitone, hexobarbitone, thiopentone; (*c*) sulphacetamide, sulphanilylurea, sulphafurazole, phthalylsulphathiazole (average error less than ±0·3%); (*d*) salts of alkaloids; (*e*) calcium salts of organic acids. Phthalylsulphathiazole was determined in the presence of sulphathiazole, and phenobarbitone in the presence of various plant extracts. J. VOLKE

219. **Polarographic determination of some water-insoluble organic substances in glacial acetic acid as solvent.** G. Dušinský and Z. Gruntová (State Inst. for Control of Drugs, Bratislava, Czechoslovakia). *Českosl. Farm.*, 1956, **5** (4), 199–201.—As a suitable solvent and an excellent extracting agent for pharmaceutical preparations, glacial acetic acid is recommended for the following substances—cinchophen, santonin and bromvaletone. A detailed procedure for tablets and suppositories is given. J. VOLKE

220. **New volumetric methods in the analysis of organic compounds. II. Determination of some aldehydes and ketones used in pharmacy.** A. Berka and J. Zýka (Inst. Anal. Chem., Charles' Univ., Prague, Czechoslovakia). *Českosl. Farm.*, 1956, **5** (1), 30–32.—The carbonyl compounds are pptd. with a soln. of 2:4-dinitrophenylhydrazine (**I**), and the excess of **I** is determined volumetrically with chloramine T as reagent. *Procedure*—The sample (1 to 10 mg) is dissolved in a small amount of ethanol (96%), and 0·01 *M* **I** in 2 *N* HCl (5 to 10 ml) is added. After 12 hr. the ppt is filtered off and washed with 2 *N* HCl (×3). The filtrate is diluted with water to twice its vol., KBr (about 1 g) is added, and potentiometric titration with 0·01 *M* chloramine T is carried out, with a platinum indicator electrode. This method has been applied to the determination of formaldehyde (in the presence of an excess of ethanol, methanol and formic acid), carvone (in caraway oil) and benzaldehyde. All the determinations may be made on a semi-micro scale. J. VOLKE

221. **Polarographic titrations of organic bases. V. The use of complex compounds of cadmium, bismuth and antimony as volumetric reagents.** V. Schillerová and J. Zýka (Inst. Anal. Chem., Charles' Univ., Prague, Czechoslovakia). *Českosl. Farm.*, 1957, **6** (2), 93–98.—For the pptn. of organic bases with polarographic indication, cadmium potassium bromide and thiocyanate among the cadmium complexes and bismuth potassium iodide among the complex salts of bismuth proved most satisfactory. No antimony salt can be used. A dropping mercury electrode was used as indicator. Complex salts of Cd and Bi form ppt. in a ratio of whole small numbers only if the titrant contains an excess of a complex-forming anion (I^-, Br^-, SCN^-). For each organic base or its salt, special conditions must be maintained. Cinchonine, narcotine and codeine hydrochlorides, and strychnine nitrate can be titrated with CdI_2 in soln. of neutral salts (0·1 *M* KNO_3, NaCl or Na_2SO_4), and the hydrochlorides of ethylmorphine and papaverine can be titrated in 0·1 *M* KNO_3. All these alkaloids with the exception of papaverine hydrochloride can be titrated in 0·1 *M* H_2SO_4. The hydrochlorides of papaverine (in 0·1 *M* KNO_3) and narcotine (in 0·1 *M* KNO_3 or 0·1 *M* H_2SO_4), arecoline hydrobromide and pilocarpine hydrochloride (in 0·1 *M* KI) can be determined by polarographic titration with $KBiI_4$. J. VOLKE

222. **Polarographic determination of individual constituents in cotarnine - narcotine and hydrastinine - hydrastine mixtures.** J. Holubek and J. Volke (Forschungsinst. für Heilpflanzen, Prague). *Pharmazie*, 1957, **12** (5), 261.—Cotarnine or hydrastinine can be determined by direct polarography in alkaline soln. Polarographic determination in alkaline soln. after oxidative hydrolysis with hot dil. HNO_3 gives the total content of cotarnine and narcotine, or of hydrastinine and hydrastine (*cf. Ibid.*, 1956, **11**, 577). The relative error is $< \pm 4\%$. A. R. ROGERS

223. **The quantitative estimation of ergotamine and ergotoxine by paper chromatography.** D. D. Jones, H. Katyama and V. E. Tyler, jun. (Coll. of Pharm., Univ. Nebraska, Lincoln, U.S.A.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1957, **46** (7), 426–427.—Under specified conditions, the R_F values of ergotamine and the ergotoxine group of alkaloids increase as a linear function of the quantity spotted on a paper chromatographic strip. This change of R_F value is used for the quant. determination of these alkaloids. *Procedure*—Prepare strips of Whatman No. 1 paper by dipping them into McIlvaine buffer of pH 4 and allowing to dry. Apply an alcoholic soln. of the alkaloid (10 to 30 μg) as a spot and develop with $CHCl_3$ saturated with 0·1 *M* citric acid soln. for 90 min. by the descending technique. Dry, examine under u.v. light, and calculate the R_F value based on the estimated front of the spot. Compare with that on a standard strip spotted with 10 μg of alkaloid. For an accuracy of ±3 μg, use the mean of four replicates each of test and standard. A. R. ROGERS

224. **Colorimetric determination of caffeine in the presence of amidopyrine and alkaloids.** Z. Gruntová (State Inst. for Control of Drugs, Bratislava, Czechoslovakia). *Českosl. Farm.*, 1956, **5** (4), 215–216.—Caffeine is pptd. from these mixtures by shaking with 0·1 *N* HCl and extracting with chloroform. The chloroform is evaporated, the residue is dissolved in 10% H_2SO_4, and caffeine is pptd. with molybdophosphoric acid. The phosphorus in the ppt. is determined colorimetrically. Amidopyrine, codeine, ephedrine and quinine do not interfere, but theophylline must be pptd. with 0·1 *N* $AgNO_3$. Results are inaccurate if papaverine is present. This method was applied to the analysis of suppositories and tablets. J. VOLKE

225. **Complexometric titrations in pharmaceutical analysis. XIV. Indirect determination of theophylline, theobromine, quinine, Analergin [antazoline] and Divascol [tolazoline].** B. Budčšínský and E. Vaníčková (Res. Inst. Pharm. and Biochem., Prague, Czechoslovakia). *Českosl. Farm.*, 1956, **5** (2), 77–80.—Theobromine, theophylline, quinine, antazoline and tolazoline are pptd. at pH 1·2 to 1·5 with bismuth potassium iodide, and the excess of reagent is determined as Bi complexometrically. Barbiturates, citrates, salicylates, ascorbic acid and phenacetin do not interfere. For theobromine, a selective method with pretreatment with mercuric potassium iodide is described which enables the determination to be made in the presence of almost all organic bases. J. VOLKE

226. **Colorimetric method for evaluating digitalis leaves.** J. Cabo Torres (Fac. de Farm., Madrid). *Inf. Quim. Anal.*, 1957, **11** (3), 69–75.—Digitalis leaves are extracted twice with 25% aq. ethanol and

the extract is treated with neutral lead acetate to remove impurities, followed by Na_2HPO_4 to remove excess of lead. The soln. is treated with alkaline picric acid and its extinction is measured, with a blue filter. A soln. of anhydrous digitoxin (0·1%) in absolute ethanol is used to construct the calibration curve. The standard deviation is ±0·00707 based on 10 determinations. G. H. FOXLEY

227. **A comparison of results estimated by the guinea-pig method and the pigeon method in the bio-assay of commercial digitoxin.** Ching-Hsi Chang, Ya-Ling Chao and Yu-Chun Hsu (The Bio-assay Dept., Central Bureau of Drug Analysis, Peking). *Acta Pharm. Sinica*, 1957, **5** (1), 73–77.—Various digitalis preparations, including digitalis leaves, tablets and tinctures and digitoxin and its injections, were assayed by both the guinea-pig method and the pigeon (lethal dose) method (B.P., 1953, p. 830), with standardised digitalis powder as reference. Results for six digitalis-leaf preparations obtained by these methods were in reasonable agreement, but for seven commercial digitoxin preparations the results obtained by the guinea-pig method were significantly lower than those obtained by the pigeon method. The difference is attributed to the inadequacy of the standard preparation of *Digitalis purpurea* employed in the assay, which contains chiefly purpurea glycosides A and B and is different in its mode of action from commercial digitoxin, which is chiefly the degraded product of purpurea glycoside A. S. H. YUEN

228. **Spectrophotometric method for the determination of cardiac glycosides. II. A spectrophotometric curve for digitoxin, acetyldigitoxin, lanatoside C and K-strophanthin.** G. A. Cinotti, G. de Simone and S. Magalini (Ist. di Clinica Med. Generale, Univ. di Roma, Italy). *G. Biochim.*, 1956, **7**, 153–158.—The method employed by Zlatkis (*J. Lab. Clin. Med.*, 1953, **41**, 486) for the determination of cholesterol has been adapted for the determination of the four glycosides possessing the same ring system as cholesterol. Equimolecular proportions of digitoxin, acetyldigitoxin, lanatoside C and K-strophanthin treated in ethanol soln. with a reagent containing 10% of Fe_2O_3 dissolved in glacial acetic acid and conc. H_2SO_4 have been examined spectrophotometrically. They showed different transmission, but all four gave the same max. at 470 mμ, compared with 560 mμ for cholesterol. P. HAAS

229. **Colorimetric determination of rutin.** J. Kucharský (Pharm. Control Inst., Prague, Czechoslovakia). *Českosl. Farm.*, 1957, **6** (2), 91–93.—A coloured product with max. absorption at 460 mμ is formed when $CrCl_2$ reacts with rutin in 40% aq. pyridine. Decomposition products, theophylline, ascorbic acid and phenobarbitone do not interfere. *Procedure for pharmaceuticals*—A sample (≡20 mg of rutin) is dissolved in 40% aq. pyridine and made up to 100 ml. A 5-ml aliquot is mixed with 40% aq. pyridine (25 ml) and then with 0·1 *N* $CrCl_2$ (1 ml), and the extinction is measured and compared with a blank. The evaluation is carried out by reference to a calibration curve. The average error is ±2%. J. VOLKE

230. **Polarography of cinnamaldehyde and its determination in cinnamon bark (Cortex cinnamomi).** G. Dušinský, M. Tyllová and Z. Gruntová (State Inst. Control Drugs, Bratislava, Czechoslovakia). *Českosl. Farm.*, 1957, **6** (2), 87–91.—In buffered soln. (pH 3·6 to 4·6), cinnamaldehyde (**I**) is reduced in three polarographic waves ($E_{\frac{1}{2}}$ = −0·86 V, −1·10 V and −1·26 V *vs.* the S.C.E. at pH 4·1). For analysis, 5% acetic acid can be used as supporting electrolyte. *Procedure*—Powdered cinnamon bark (approx. 0·5 g) is extracted in an air-tight bottle with 25% acetic acid (**II**) (2 × 20 ml) for two 1-hr. periods in a mechanical shaker. The extracts are filtered through paper and made up to 50 ml with **II**. A 2-ml aliquot is mixed with 25% **II** (2 ml) and H_2O (16 ml) and a polarographic curve is recorded after removal of oxygen. A 2 mg-% soln. of **I** in 5% **II** is used as standard. J. VOLKE

231. **Paper chromatography of the tetracycline antibiotics and their epimers.** G. B. Selzer and W. W. Wright (Food Drug Admin., Washington, D.C., U.S.A.). *Antibiot. & Chemother.*, 1957, **7** (6), 292–296.—Ascending chromatography is carried out on Whatman No. 1 paper moistened with pH 3·5 buffer (0·1 *M* citric acid plus 0·2 *M* Na_2HPO_4). Excess of buffer is removed by blotting, and 2 μl of sample solution in methanol (1 mg per ml) applied. After allowing the paper to dry partially for a few min. in air, it is developed with a mixture of $CHCl_3$ - nitromethane - pyridine (10:20:3) until the solvent front rises 4 in. The paper is then allowed to dry and examined under u.v. light for fluorescence (enhanced by NH_3 vapour). Appropriate spots are cut out, extracted with phosphate buffer (pH 4·5) and determined by a cylinder-plate method. Details are given for the determination of tetracyclines in animal feeds and for quatrimycin (the inactive epimer of tetracycline) in pharmaceutical preparations. W. H. C. SHAW

232. **Determination of salicylic acid in aspirin.** C. W. Strode, jun., F. N. Stewart, H. O. Schott and O. J. Coleman (Monsanto Chem. Co., St. Louis, Mo., U.S.A.). *Anal. Chem.*, 1957, **29** (8), 1184–1186.—A spectrophotometric and a visual method are described for the determination of free salicylic acid in aspirin, based on the formation of the ferric salicylate colour. By controlling the variables, which may affect the colour, and the hydrolysis of the aspirin, precision and accuracy within ±0·0005% can be attained. The hydrolysis correction is predetermined and is applicable for concn. of up to ≃0·25% of free salicylic acid in crystalline acetylsalicylic acid and in starch - aspirin formulations. The visual method is limited to essentially colourless samples. G. P. COOK

233. **Polarographic distinction between Bromisoval [bromvaletone] and Bromadal [carbromal] and their determination.** V. Volková (Polarographic Inst. Acad. Sci., Prague, Czechoslovakia). *Českosl. Farm.*, 1956, **5** (4), 203–206.—For the polarographic determination of bromvaletone (**I**) and carbromal (**II**), supporting electrolytes of 0·1 *M* HCl or acetate buffers with approx. 20% ethanol are used. The reduction waves of both substances coincide when one substance is determined in the presence of the other, and the composite wave (corresponding to the sum of both compounds) is recorded. **I** alone is then determined, after hydrolysis in 0·5 *M* NaOH, as α-bromo*iso*valeric acid; the polarographically inactive hydroxydiethylacetic acid is formed from **II**. The difference between both determinations gives the amount of **II**. J. VOLKE

234. **Microchemical detection of barbiturates with copper - ammonia reagent.** H. M. Romijn. *Pharm. Weekbl.*, 1957, **92** (12), 397–406.—Barbiturates form blue and/or violet crystals with soln. of Cu acetate, 1 and 10%, in 25% aq. NH_3; some brownish-yellow, or colourless, crystals may also be formed. The 15 barbiturates examined fell into three categories—(*i*) those forming both blue and violet crystals, (*ii*) those forming blue, but no violet, crystals, (*iii*) those forming, after heating, either violet or colourless, but no blue, crystals. The reagent is not specific for barbiturates and it reacts with many organic acids and most sulpha compounds. W. J. WRIGHT

235. **Polarography of urea and thiourea derivatives. IX. Determination of thiobarbituric acid derivatives and of 4-methyl-2-thiouracil in pharmaceutical preparations.** O. Manoušek and P. Zuman (Polarographic Inst., Acad. Sci., Prague, Czechoslovakia). *Českosl. Farm.*, 1956, **5** (4), 193–195.—Anodic polarographic waves of thiobarbiturates (in 0·1 *M* NaOH) and of 4-methyl-2-thiouracil (in *M* NaOH) were used for determinations in pharmaceutical preparations. The method is more specific than the usual titrations, and considerably more rapid. The average error is ±3%. J. VOLKE

236. **Polarographic determination of bromocholine (Kathesin).** G. Dušinský (State Inst. for Control of Drugs, Bratislava, Czechoslovakia). *Českosl. Farm.*, 1956, **5** (4), 202–203.—Bromocholine bromide (**I**) gives a polarographic reduction wave in soln. of $Na_2B_4O_7$ ($E_{\frac{1}{2}} = -1{\cdot}06$ V) which is suitable for analytical purposes. *Procedure*—A sample containing 5 to 25 mg of **I** is dissolved in H_2O (50 ml), and 1% basic fuchsine (1 ml) and 0·05 *M* $Na_2B_4O_7$ (10 ml) are added. After removal of O_2 the polarogram is recorded. Evaluation is made with the use of a calibration curve. Chlorides and theobromine do not interfere. J. VOLKE

237. **Photometric estimation of *p*-aminobenzoic acid in presence of procaine hydrochloride in drug preparations. II.** J. Richter (State Inst. for Drug Testing, Berlin). *Arzneimittel-Forsch.*, 1957, **7** (3), 200–201.—The earlier method (*cf. Anal. Abstr.*, 1955, **2**, 1312) is improved by the use of a 5% aq. soln. of $Na_2HPO_4.2H_2O$ instead of NaOH before extraction of the procaine hydrochloride (**I**) with $CHCl_3$. Errors introduced by varying times of contact with the alkaline soln. are thus obviated (treatment time may vary between 1 and 6 min. without appreciable effect on the results). *Procedure*—The 2-ml sample of soln., containing ≯0·5% of **I**, is treated with 5 ml of the phosphate soln. in a small separating funnel, then shaken successively with 20- and 10-ml portions of $CHCl_3$. The aq. phase is freed from $CHCl_3$ by centrifuging for about 1 min., then a 5-ml portion is mixed with 5 ml of *N* HCl and 5 ml of freshly prepared 0·5% $NaNO_2$ soln. After 2 min., 10 ml of a 0·1% soln. of thymol (in 5% aq. NaOH) is added and the soln. is made up to 100 ml. The extinction of the resulting yellow - orange soln. is determined after 2 min. with a Pulfrich photometer and S47 filter. The sensitivity is high; 0·0025% of *p*-aminobenzoic acid can be detected in a 0·5% soln. of **I**. H. K. WHITEHEAD

238. **Photometric study of the colour reaction between phenazone and ferric iron.** J. Čelechovský and V. Krejčí (Masaryk Univ., Brno, Czechoslovakia). *Českosl. Farm.*, 1957, **6** (2), 98–103.—Phenazone (**I**) reacts with Fe^{3+} to form an orange - red complex, whose max. absorption is at 470 mμ. The dissociation and equilibrium constants have been determined. For the photometric determination of **I**, a soln. of pH 2 and 5×10^{-2} *M* Fe^{3+} is recommended. Aromatic amines (anaesthetics), codeine, phenacetin and sugars do not interfere. As little as 0·1 to 30 mg of **I** in 50 ml can be determined. J. VOLKE

239. **Colorimetric determination of 1-*p*-hydroxyphenyl-2-methylaminopropanol [hydroxyephedrine].** M. Voldan (State Inst. for Control of Drugs, Prague, Czechoslovakia). *Českosl. Farm.*, 1956, **5** (3), 146–147.—*p*-Hydroxyephedrine reacts with Millon's reagent to yield a coloured product. The extinction is measured at 500 mμ and evaluated by means of a calibration curve (average error ±4%). J. VOLKE

240. **Determination of 4-amino-6-hydroxy*iso*phthalic acid and of *m*-aminophenol in sodium *p*-aminosalicylate.** K. A. Bakker (Lab. voor Artsenijbereidkunde, Amsterdam, Holland). *Pharm. Weekbl.*, 1957, **92** (7), 205–213.—Extinction values at 239 mμ and a calibration graph are given for the determination of 4-amino-6-hydroxy*iso*phthalic acid in the drug in ethanolic (96%) soln. The presence of 0·25% of this impurity can be detected. Similar data are given which allow of the determination of *m*-aminophenol in the aq. diazotised soln. of the drug by spectrophotometric measurement at 274 mμ. The presence of 0·5% of this impurity can be detected. P. S. ARUP

241. **Colorimetric determination and paper chromatography of the calcium salt of *p*-benzamidosalicylic acid.** J. Vacek and M. Lašťovková (State Inst. for Control of Drugs, Prague, Czechoslovakia). *Českosl. Farm.*, 1956, **5** (2), 75–77.—The method is based on the formation of a coloured complex of *p*-benzamidosalicylic acid (**I**) with Fe^{3+}. Pulfrich filter S53 is used for measurements, and a calibration curve is used for evaluation. Extraction from tablets is made with 65% ethanol. The average error is ±2%. As little as 0·5 to 5 μg of salicylic acid and *m*-aminophenol in 200 to 250 μg of **I** can be determined semi-quantitatively on Whatman paper No. 1 with the system pyridine - *n*-butanol - satd. KCl - 25% aq. NH_3 (6:6:5:3). J. VOLKE

242. **Polarographic determination of some esters of nicotinic acid.** J. Volke (Polarographic Inst. Acad. Sci., Prague, Czechoslovakia). *Českosl. Farm.*, 1956, **5** (4), 206–208.—Esters of nicotinic acid can be determined by using the reduction wave at pH 4·7 or, if rapid analysis is possible, in alkaline soln., *e.g.*, at pH 9·7 for the methyl ester. Soln. for the determination of the *iso*amyl and benzyl esters should contain ethanol. Results are evaluated according to a calibration curve or by standard addition. The accuracy is ±2%. Free nicotinic acid does not interfere. Esters and free acid can be determined simultaneously. J. VOLKE

243. **Determination of Ethiacin (2-hydroxyethyl nicotinate).** B. Kakáč and Z. J. Vejdělek (Res. Inst. Pharm. and Biochem., Prague, Czechoslovakia). *Českosl. Farm.*, 1956, **5** (3), 140–146.—Commercial 2-hydroxyethyl nicotinate (**I**) contains nicotinic acid (**II**) and dinicotinyloxyethane (**III**) as impurities. **I** is determined polarographically at pH 10·2 in the presence of **II** and **III**. The free **II** is determined by alkalimetric titration, and **III** polarographically at pH 10·2 after removal

of **I** and **II**, use being made of their different solubilities in alkaline soln. As little as 0·1% of **III** was determined by this method. J. VOLKE

244. **New volumetric method for the determination of isonicotinic acid hydrazide [isoniazid].** P. Spacu and Gr. Teodorescu (Lab. Inst. Politehnic, Bucharest). *Rev. Chim., Bucharest*, 1957, **8** (1), 42–43.—The method is based on the treatment of isoniazid (**I**) with Reinecke's salt (**II**) to give a cryst. lilac ppt. of a complex $[C_5H_4NCONH{\cdot}NH_3][Cr(SCN)_4(NH_3)_2]$, which is slightly sol. in water, rather sol. in alcohol and ether, and completely sol. in acetone; it is decomposed when heated in a drying oven. For the determination, the ppt. is dissolved in acetone, and excess of $AgNO_3$ is added, followed by back-titration with NH_4SCN, with ferric alum as indicator. *Procedure*—A soln. of **I** (5 to 10 ml containing 0·025 to 0·05 g) is acidified with three drops of dil. H_2SO_4, and diluted with 20 ml of water. A freshly prepared 2% soln. of **II** is added dropwise, with continual shaking. After the pptn., the soln. is an intense red. Immediately the ppt. has settled, it is filtered off, washed 3 or 4 times with 2 to 4 ml of **II** soln. (0·1%), and twice with 0·5 ml of water, taking care that none dissolves. The ppt. is dissolved from the crucible with acetone, both crucible and flask being washed with water, and the soln. is collected in a 100-ml flask. An excess (10 to 15 ml) of 0·1 *N* $AgNO_3$ is added, and Ag reineckate is pptd. A few drops of HNO_3 are added to flocculate the ppt., and the soln. is made up to 100 ml with water. The ppt. is filtered off, and an aliquot portion (25 to 50 ml) of the filtrate is acidified with a few drops of HNO_3; after the addition of 2 ml of ferric alum, the filtrate is titrated with 0·1 *N* NH_4SCN. H. SHER

245. **Comparison of three volumetric methods for the determination of Tibione [thiacetazone].** Yu-Chung Chang, Chi-Li Wang and Suh-Lan Woo (School of Pharmacy, Peking Medical College). *Acta Pharm. Sinica*, 1957, **5** (1), 17–22.—Volumetric determinations of thiacetazone, both pure and in tablets, were compared, the argentimetric method (Middeldorf, *Süddtsch. ApothZtg*, 1950, **90**, 804), the iodimetric method (Sandri, *Brit. Abstr. C*, 1952, 99) and the ferric chloride method (Wollenberg, *Brit. Abstr. C*, 1952, 452) being used. All three methods gave accurate results. For pure samples the iodimetric method was most suitable because of its rapidity and sensitivity, but for tablets the ferric chloride method is recommended.
S. H. YUEN

246. **Determination of 4-*n*-butyl-1:2-diphenylpyrazolidine-3:5-dione [phenylbutazone].** F. Jančik, E. Kraus, B. Buděšínský and O. Činková (Res. Inst. Pharm. and Biochem., Prague, Czechoslovakia). *Českosl. Farm.*, 1957, **6** (2), 105–108.—The method is based on the addition of Br to the double bond of the enol form of phenylbutazone (**I**). *Procedure*—The sample (200 to 300 mg of **I**) is dissolved in glacial acetic acid (30 ml), HCl (5 ml) and 20% KBr (5 ml), and titrated with 0·1 *N* $KBrO_3$. The end-point is determined potentiometrically. A semi-micro determination can be carried out with 0·01 *N* $KBrO_3$. If an excess of 0·1 *N* $KBrO_3$ (25 ml) is added, the free Br is removed by 1% aniline soln. (5 ml) and, after 5 min., KI (1 g) is added, and the $KBrO_3$ is determined by titration with 0·1 *N* $Na_2S_2O_3$ (starch as indicator). Before the end-point is reached, the soln. should be diluted to 50 ml with H_2O. An acidimetric titration with ethanolic 0·1 *N* NaOH (to phenolphthalein) is possible. J. VOLKE

247. **Polarographic determination of arecoline-acetarsol (Nemural Hoechst) in tablets.** J. Blažek (State Inst. for Control of Drugs, Prague, Czechoslovakia). *Českosl. Farm.*, 1956, **5** (4), 208–209.—Nemural Hoechst is determined polarographically in 0·1 *N* LiCl. J. VOLKE

248. **Identification of drugs. II. Sulphonamides.** H. Baggesgaard-Rasmussen, J. Berger and G. Espersen (Royal Danish School of Pharmacy, Copenhagen). *Dansk Tidsskr. Farm.*, 1957, **31**, 53–68.—Detailed directions are given for the prep. and purification of the N^4-acetyl derivatives of 12 sulphonamides and of the amines obtained by the hydrolysis [with 40% (v/v) H_2SO_4] or pyrolysis of the sulphonamides. Tabulated data include the m.p. and equiv. wt. of the acetyl derivatives and the m.p. and other information on the amines. The m.p. are often higher than those given in the literature. For the prep. of the amines, preference is given to the method of hydrolysis, which can also be applied to the investigation of the composition of the acetyl derivatives. P. S. ARUP

249. **Determination of sulphonamides by polarographic titration.** D. Rybář and V. Skřivan (Chemopharma, Anal. Lab., Ustí n.L., Czechoslovakia). *Českosl. Farm.*, 1956, **5** (3), 147–149.—Various sulphonamides were titrated with 0·1 *M* $NaNO_2$ (in 12·5% HCl) as reagent. A platinum rotating or stationary electrode is used as indicator electrode, and satd. calomel, graphite or platinum as reference electrodes. The electrodes are connected directly across a galvanometer (except the platinum reference electrode where a potential difference of 0·4 V is necessary). The average error is ±0·5%.
J. VOLKE

250. **Determination of 6-mercaptopurine.** F. Jančik, B. Buděšínský and O. Činková (Res. Inst. Pharm. and Biochem., Prague, Czechoslovakia). *Českosl. Farm.*, 1957, **6** (2), 108–110.—6-Mercaptopurine (**I**) can be titrated either (*a*) bromimetrically or (*b*) iodimetrically, the SH group being oxidised in both methods. *Procedure*—(*a*) Dissolve **I** (30 to 40 mg) in 6 *N* H_2SO_4 (20 ml), add exactly 25 ml of 0·1 *N* $KBrO_3$ and set aside in the dark for 15 min. Then add KI (1 g) and after 5 min. titrate the liberated iodine with 0·1 *N* $Na_2S_2O_3$, with starch as indicator. (*b*) Dissolve **I** (50 to 100 mg) in twice-distilled H_2O (15 ml) and 5 *N* NaOH (10 ml). Then titrate with 0·1 *N* iodine, with potentiometric indication. Hypoxanthine does not interfere. Both methods are accurate to within ±0·3%, and are recommended for inclusion in pharmacopoeias.
J. VOLKE

251. **Volumetric determination of 6-mercaptopurine.** J. Blažek and Z. Stejskal (State Inst. for Control of Drugs, Prague, Czechoslovakia). *Českosl. Farm.*, 1956, **5** (1), 29–30.—Silver nitrate forms with 6-mercaptopurine (**I**) in aq. NH_3 soln. an almost insoluble ppt. *Procedure*—Finely powdered tablets (approx. 5 tablets corresponding to 250 mg of **I**) are dissolved in H_2O (50 ml) and 10% aq. NH_3 (15 ml); 0·1 *N* $AgNO_3$ (50 ml) is added and the vol. is made up to 250 ml with H_2O. The soln. is filtered through a filter-paper, the first 20 ml of the filtrate is discarded, and in a later aliquot (50 ml) the excess of Ag^+ is determined by titration with 0·1 *N* NH_4SCN. The average error is ±0·25%.
J. VOLKE

252. **Polarographic determination of some derivatives of phenothiazine important in pharmacy.** J. Blažek (State Inst. for Control of Drugs, Prague, Czechoslovakia). *Českosl. Farm.*, 1956, **5** (4), 210–211.—Phenothiazine and its derivatives react with tungstosilicic acid in 3·5% HCl to give ppt. of low solubility. If 0·01 *M* tungstosilicic acid is used, 20 to 50 mg of the sample may be titrated polarographically (average error ±4%). Phenothiazine, diethazine, chlorpromazine and promethazine were determined by this method, some of them in tablets. J. VOLKE

253. **Isolation and quantitative determination of synthetic anti-malarials by ion-exchange techniques.** R. Ravel (State Univ., Iowa City, U.S.A.). *Dissert. Abstr.*, 1956, **16** (10), 1889–1890.—A technique for the assay of synthetic anti-malarial org. bases is described. S.C.I. ABSTR.

254. **Colorimetric determination of menadione [menaphthone] sodium bisulphite and its preparations.** Yuan-Yau Chou and Ju-Cheng Hsü (Wu-Han Municipal Drug Bureau, Wu-Han, China). *Acta Pharm. Sinica*, 1957, **5** (1), 29–37.—The application of Craven's colour reaction (*J. Chem. Soc.*, 1931, 1605) of *p*-benzoquinones with ethyl cyanoacetate to menaphthone was examined with reference to the effect of varying conditions, such as amounts of reagents, temp., time and interference by some inorganic salts. A simple accurate colorimetric method for the determination of menaphthone sodium bisulphite (**I**) was developed. To 10 ml of 55% ethanolic soln., containing 0·5 to 0·8 mg of **I**, add 0·5 ml of an ethanolic soln. of ethyl cyanoacetate (1:20) and 2·5 ml of aq. NH_3 (sp. gr. 0·89). Dilute with ethanol to 25 ml after 5 min. and mix well. Allow to stand for 30 min., read the extinction of the blue colour at 593 mμ against H_2O in a spectrophotometer, or in a photo-electric colorimeter with a yellow filter. This method is suitable for quant. estimation of **I** in injections and tablets with an accuracy of ±1%. S. H. YUEN

255. **[Use of] tetrazolium salts. I. Determination of 2-methyl-1:4-naphthaquinone [menaphthone].** M. Smíd, B. Kakáč and Z. Pádr (Res. Inst. Pharm. and Biochem., Prague, Czechoslovakia). *Českosl. Farm.*, 1956, **5** (4), 212–215.—Products formed by the action of tetramethylammonium hydroxide on menaphthone reduce tetrazolium salts to coloured formazans, which can be determined spectrophotometrically. The method is used for the determination of the tetrasodium salt of 2-methyl-1:4-naphthaquinone phosphate. The phosphate can be determined colorimetrically after reaction with molybdate. J. VOLKE

256. **The determination of cyanides in bitter almond water.** M. Šaršúnová (Control Lab. Medica, Bratislava, Czechoslovakia). *Farmácia*, 1956, **25** (7), 196–197.—A modification of the complexometric determination of cyanides (*cf.* Huditz and Flaschka, *Z. anal. Chem.*, 1952, **136**, 185) is described. *Procedure*—To the sample (25 g) add aq NH_3 (10%) (2 ml), dilute with water (100 ml) and titrate with 0·1 *M* $NiSO_4$, with murexide as indicator (colour change, violet to yellow). J. ZÝKA

257. **Complexometric determination of calcium carbonate in [compound] sulphur ointments.** M. Šaršúnová (Control Lab. Medica, Bratislava, Czechoslovakia). *Českosl. Farm.*, 1956, **5** (4), 217–218.—After extraction by boiling with conc. HCl, and making alkaline with 20% NaOH (pH 12), the diluted soln. is titrated with 0·1 *M* EDTA (disodium salt), with murexide as indicator, to a blue - violet colour. J. VOLKE

See also Abstracts 69, 127, 183, 184, 185, 308.

Food

258. **Lead error in the polarisation of British raw beet sugars.** L. Eynon, A. E. Tate, J. F. T. Oldfield and J. G. N. Gaskin (7–8, Idol Lane, London). *Int. Sugar J.*, 1957, **59**, 38–39.—The method of lead error determination is fully described for tests on ten samples of raw sugar from four beet factories. The lead sub-acetate reagent, both in wet and dry forms, was prepared as for the corresponding error determinations on raw cane sugar (*cf. Anal. Abstr.*, 1957, **4**, 3749). Results are tabulated. Although significant (P = 0·01), the effect of the reagent on raw beet sugar polarisation is small, especially with the dry lead. Regression analyses for both sets of data were prepared, and correction tables for pol 93 to 99 are reproduced. The fiducial limits of the corrections are given at the 95% inclusive level. The dry lead correction for raw sugars whose pol is 96 to 98 is less than the possible error of that correction (since it is near the middle of the fiducial range) and such corrections therefore have little value. The effects of wet and dry lead on beet pol are smaller than the corresponding effects on cane sugar, probably owing to the different relative proportions and composition of optically active non-sucrose substances after pptn. or formation of lead complexes. The effect of basicity of the lead acetate on these corrections is being investigated. In view of the small corrections, the 15th Meeting of the British National Committee of I.C.U.M.S.A. decided to make no recommendations regarding the application of such corrections to raw beet sugars. SUGAR IND. ABSTR.

259. **Flour testing. I. A comparison of the Brabender extensograph, Chopin alveograph and Simon extensometer methods of testing bread flours with particular reference to the effect of various forms of flour treatment.** R. Bennett and J. B. M. Coppock (Brit. Baking Ind. Res. Ass., Chorleywood, England). *J. Sci. Food Agric.*, 1956, **7** (12), 754–764.—A critical account of methods for testing bread flours with three instruments in common use is given, with the experimental requirements for obtaining dependable results. The Simon extensometer is the most suitable instrument, but the Brabender technique can be adapted to the standard test conditions. For reliable comparison of samples, it must be ensured that the dough contains the right amount of water to produce the correct consistency at the end of the fermentation, that yeast is included, that a resting period of 3 hr. is allowed before final moulding, and that adequate moulding is given. D. G. FORBES

260. **The study of Matweef's method for the detection of flour from hard wheats in semolinas and flour products.** M. R. Alliot (Lab. Central de la Répression des Fraudes, Paris, France). *Ann. Falsif.*, 1957, **50**, 116–124.—Improvements are suggested for the method of Matweef (*Compt. Rend., Acad. Agric. France*, 1952, **39**, 16), in which the estimation of steroid content provides a measure of the proportion of hard wheat used in the preparation of various flour products. (*a*) Grinding is effected

by a hammer mill which will handle a 2-kg sample. (*b*) The steroids are extracted from 100 g of the sample with 200 ml of acetone. (*c*) The extract is maintained at a temp. of $-5° \pm 1°$ for $\leqslant 12$ hr. to ensure complete pptn. (*c*) Because of the transience of the emerald-green colour developed in the test, this is allowed to pass to a clear yellow-brown (24 to 36 hr.), which shows greater stability. (*e*) To obviate the necessity of repeated cleaning of the cells, an absorptiometer adapted for the use of test-tubes with ground glass stoppers was employed. Fair agreement was obtained between the values for the steroid content of semolinas and of the hard wheat from which the semolinas were manufactured.

D. G. FORBES

261. **Determination of nitrite and nitrate [in meat-curing brines] by a polarographic process.** P. Spanyár, E. Kevei and M. Kiszel (Forschungsinst. für Konserven-, Fleisch- und Kälteindustrie, Budapest). *Acta Chim. Acad. Sci. Hung.*, 1957, **11** (3–4), 329–337.—Earlier methods are slow and cumbersome and liable to give anomalous results in the presence of compounds containing amino groups. In the present method, the total NO_2^- plus NO_3^- is determined polarographically in the presence of ethanol, which precipitates any proteins that might be present, *e.g.*, in meat-curing brines. To a further sample HCl is added, and the HNO_2 so liberated forms ethyl nitrite, which can easily be removed in a stream of inert gas. The remaining NO_3^- can then be estimated polarographically and the NO_2^- obtained by difference. Soln. containing 50 to 150 μg of NO_2^- plus NO_3^- in 0·5 ml can be estimated with an accuracy of $\pm 8\%$. F. J. M.

262. **The acidity of formalised milks and the measurement of lactic acid produced at the expense of lactose.** I. S. Lascano Ruiz and Y. Prat (Lab. Municip., Paris, France). *Ann. Falsif.*, 1957, **50**, 101–107.—The official (French) method for preservation of samples of milk by the addition of small amounts of formaldehyde and paraformaldehyde induces a slight increase of the acidity as measured by standard procedures. Fresh milk preserved by this means immediately acquires a pH value of $\simeq 6·5$, and titration with phenolphthalein as indicator gives a figure which includes the induced acidity. The indicator cannot therefore be used in the direct titration of lactic acid in preserved milks, but a correct value can be obtained potentiometrically or by the use of indicators such as chlorophenol red or bromocresol purple, which give end-points at pH 6·5 to 6·6. D. G. FORBES

263. **Determination of diphenyl in citrus fruits.** H. Böhme and L. Bertling (Inst. für Pharm. Chemie und Lebensmittelchemie, Univ. Marburg a.d. Lahn, Germany). *Z. LebensmittUntersuch.*, 1957, **105** (4), 311–318.—The procedure consists in the steam-distillation (45 min.) of the orange or lemon peel (100 to 150 g), cut into small pieces in water, in a special apparatus of measured proportions, in the presence of *cyclo*hexane, which extracts the diphenyl and volatile oil. The *cyclo*hexane solution is shaken with conc. H_2SO_4 (3 to 5 times), separated, washed, and dried over Na_2SO_4 and, after a final treatment with an "acid" alumina column, the extinction is read at 250 mμ. S.C.I. ABSTR.

264. **Analysis of fats from chocolate products (and cacao beans). II.** F. T. van Voorst (Keuringsdienst voor Waren, Alkmaar, Holland). *Chem. Weekbl.*, 1957, **53** (32), 443–445.—In a continuation of a previous paper (*Anal. Abstr.*, 1957, **4**, 3463), the iodine values of the solid fatty acids of hardened fats used in the manufacture of chocolate have been determined; the values obtained for such fats produced from palm-kernel, fish and arachis oils varied from 39·8 to 54·0. Corresponding values for a variety of 40 samples of chocolate preparations and beans gave an average of 1·7, including a few samples with values as high as 4·5 to 5·8, indicating the presence of hardened fats. Four samples of chocolate deliberately prepared with such fats gave values from 4·2 to 10·0. Determination of the ratio 0′ to I′ for cacao beans, in which 0′ and I′ represent the critical dissolution temp. and the iodine value, respectively, of milk-free fat, gave an average value for 19 different samples of cacao beans of 1·10 compared with 1·13 for 33 samples of chocolate and 1·11 for 13 samples of milk chocolate, with a probable error of 0·4 for all these. The value for cacao butter was 1·13 with a probable error of 0·2. P. HAAS

265. **Tetrabromide method for estimating linoleic acid in fatty acid mixtures.** K. D. Phatak and J. S. Aggarwal (Nat. Chem. Lab., Poona). *J. Sci. Ind. Res., B, India*, 1957, **16**, 19–22.—*Procedure*—Linoleic acid (1 g) or fatty acid mixture (enough to give 0·8 to 1·2 g of tetrabromostearic acid) is weighed into a 50-ml test-tube and dissolved in light petroleum (30 ml). The soln. is cooled to between 0° and 5° and Br added dropwise until the soln. becomes orange; it is then kept at the same temp. for 3 to 4 hr. The ppt. is transferred to a sintered glass crucible with the aid of light petroleum and dried to constant weight, first at 60° and then in a vacuum desiccator. The tetrabromide value is given by (wt. of ppt. × 100)/(wt. of fatty acid), from which the percentage of linoleic acid can be calculated. Results agree, to within $\pm 3\%$, with those obtained by the spectroscopic method.

C. A. SLATER

266. **Determination of foots in raw linseed oil.** P. J. de Coninck and J. Delacourt (Belgian Congo Refineries, Baasrode). *Chim. Peint.*, 1956, **19** (12), 445–461.—The oil (200 ml) is treated with a flocculating agent (H_2O - 65% HNO_3) (3:1) (1·4 ml), then shaken for 30 min. at $\geqslant 25°$, cooled to between 15° and 17°, transferred to 100-ml tubes, and centrifuged for 30 min.; the vol. of foots is then read. The H_2O content of the original oil is determined from the loss in wt. on heating for 2 hr. at 105° to 110°. The vol. % of foots is then corrected to zero H_2O content by means of calibration graphs to give the vol. % of foots M.B.B. (Méthode Belge Baasrode). The influence of the amount of flocculating agent, temp., H_2O content, free acidity and neutralisation, and the reproducibility have been examined. After the flocculation the oil gives no ppt. with H_2O, but a second flocculation with the reagent gives a further ppt. of 1 to 1·5%. After the first treatment the oil still contains an appreciable proportion of its P and ash content and this is not appreciably affected by the second treatment. Refined oils which show a zero foots content by the method still contain a small amount of P.

L. A. O'NEILL

267. **Application of conversion factors for the determination of vitamin A in fish-liver oils.** S. Balasundaram, H. R. Cama, P. R. Sundaresan and T. N. R. Varma (Dept. of Biochem., Indian Inst. of Sci., Bangalore). *J. Sci. Ind. Res., C, India*, 1957, **16**, 8–11.—It is found that, in the spectrophotometric analysis of fish-liver oils for vitamin-A

potency, the $E^{1\%}_{1cm}$ at 328 mμ (gross) × 1600 value does not agree with the $E^{1\%}_{1cm}$ at 328 mμ (corrected) × 1910 (*cyclo*hexane) value, when the oils become oxidised during storage. $E^{1\%}_{1cm}$ at 290 mμ × 3000 (*cyclo*hexane) gives an approx. value for the original potency of the oil. C. A. SLATER

268. **Chromatographic separation of vitamins A and D with the aid of kaolin as a rapid quantitative method.** H. Tschapke and H. Plessing (Anstalt Vitaminforsch. Vitaminprüfung, Potsdam-Rehbrücke). *Pharmazie*, 1957, **12** (5), 262–264.—Vitamin-A alcohol is quantitatively removed from $CHCl_3$ soln. by kaolin that has been activated by boiling for 15 min. with 2 *N* HCl and washing with hot water until neutral. This is the basis of a method for the determination of vitamin D (**I**) in the presence of vitamin A (**II**) in pharmaceutical products other than fish-liver oils. The results agree well with those obtained biologically, and are accurate in the presence of up to ten times as many units of **II**. *Procedure*—Saponify the sample by refluxing with 2 *N* alcoholic KOH, extract the unsaponifiable matter with ether, wash, dry and evaporate to dryness. Dissolve the residue in alcohol-free $CHCl_3$ and add activated kaolin until no further blue colour (due to **II**) is given. Filter through sintered glass (3G3) and wash the residue well. Dilute the filtrate to a concn. of ≃300 units of **I** per ml and determine by the $SnCl_2$ method previously described (*cf. Anal. Abstr.*, 1956, **3**, 1161). A. R. ROGERS

269. **The ultra-violet spectrum of vitamin A_2.** G. Lambertsen and O. R. Brækkan (Gov. Vitamin Lab., Norwegian Fisheries Res. Inst., Bergen). *Acta Chem. Scand.*, 1957, **11** (3), 575–576 (in English).—The u.v. absorption curve method has previously been shown to give good results in estimating vitamin A_2 and all-*trans* vitamin A_1. Samples of vitamin A_2 from perch liver, which was found to contain no vitamin A_1 or *cis* isomers, were examined by u.v. absorption. Results obtained were comparable with those of Cama and Morton (*Analyst*, 1953, **78**, 74) on vitamin A_2 made from retinene$_2$, but the ratio of extinction values suggested a higher degree of purity. F. J. M.

270. **Reversible and irreversible processes at the dropping electrode. V. Thiamine.** J. Sancho and P. Salmerón (Lab. Quim.-Fis. Electr., Univ. Murcia, Spain). *An. Real Soc. Esp. Fis. Quim., B*, 1957, **53** (2), 117–128.—The oscillopolarographic behaviour of thiamine in 0·1 *N* NaCl and KCl soln., buffered and unbuffered, shows that a cathodic wave ($E_{\frac{1}{2}} = -1{\cdot}26$ V *vs.* the S.C.E.) appears in unbuffered soln., and is influenced by the hydrogen wave at low pH. Slightly above pH 9 there exists an anodic wave ($E_{\frac{1}{2}} = 0{\cdot}17$ V) and a further cathodic wave due to reduction of –S– at pH 9·1. The combined wave reaches a max. at pH 9·86 and decreases at pH 12; it is steady ($E_{\frac{1}{2}} = 0{\cdot}40$ V) for pH values of 9·6 to 10·2, during the passage of air, so that it may be used for quant. determination. Lower quant. limits are, for the cathodic wave in 0·1 *N* KCl, 2×10^{-4} *M*; in 0·1 *N* NaCl, $1{\cdot}6 \times 10^{-4}$ *M*, both unbuffered; for the anodic - cathodic wave in 0·1 *N* KCl, 3×10^{-4}; in 0·1 *N* NaCl, $1{\cdot}5 \times 10^{-4}$, both at pH 9·86. The anodic wave is reversible in both soln. at pH 9·86 but the other waves are both irreversible. G. F. PENNY

271. **New method for the determination of ascorbic acid.** F. Moreno Martín and F. Hernández-Gutiérrez (Fac. Pharm., Barcelona, Spain). *An. Bromatologia*, 1956, **8** (3), 261–265.—The method is based on the reduction of Denigès soln. by ascorbic acid in the cold to give a mixed ppt. of Hg_2SO_4 and Hg, which is separated and determined iodimetrically. The sample soln. (2 to 5 ml containing 0·1 to 1·25 g of ascorbic acid per 100 ml) is added to the same vol. of reagent (0·23 *M* $HgSO_4$ in 3·3 *M* H_2SO_4), mixed and set aside for 30 min. The mixture is filtered through a G4 sintered crucible, the ppt. is washed with water (×3) and then dissolved in 10 ml of 0·1 *N* iodine to which is added 1 g of KI. The soln. is passed through the filter into a clean vessel, washing with 1-ml portions of 20% KI soln. until the filtrate passes through colourless. The excess of iodine in the filtrate is titrated with 0·05 *N* $Na_2S_2O_3$. When applied to pure aq. soln. of ascorbic acid, the method has a maximum error of 1%. E. C. APLING

See also Abstracts 139, 160.

Sanitation

272. **Micro-sampling method of determining gases and vapours, particularly halogenated hydrocarbons, in air.** F. Call (Imperial Coll. Field Sta., Sunninghill, Berks., England). *J. Appl. Chem.*, 1957, **7** (5), 210–215.—An apparatus for drawing an accurately measured sample (1 to 2 ml) of air through a heated silica combustion tube and then through an absorption vessel is described. The Br produced by the thermal decomposition of brominated hydrocarbons in the sample is estimated by absorptiometric measurement of the colour change of a soln. of phenol red in phosphate buffer. Bromomethane and 1:2-dibromoethane may be estimated in the range 0·5 to 170 μg per ml with a standard deviation of about 0·5%. F. J. M.

273. **Tentative method for determining volatile - non-volatile ratios of aerosol formulation. Vacuum distillation method.** Chemical Specialties Manufacturers Ass. (110 East 42 Street, New York). *Soap, N.Y.*, 1957 *Blue Book*, 238, 269.—Samples, in triplicate, are withdrawn from the aerosol container through a needle-valve into weighed vacuum flasks. The propellent is allowed to boil off through the side arm by placing the flask in a water bath at 120° F. When boiling subsides, vacuum is gently applied and continued until the flask attains const. wt. The amount of sample withdrawn is ≃50 g. S.C.I. ABSTR.

274. **Determination of atmospheric mercury trapped in permanganate solutions: a modified method.** R. G. Drew and E. King (M.R.C. Lab., Holly Hill, Hampstead, London). *Analyst*, 1957, **82**, 461–467.—A known vol. of air is drawn through acid $KMnO_4$ soln. and the mercury is extracted with dithizone soln. after reduction of the $KMnO_4$. In the method described, H_2O_2 is used as the reducing agent and gives more reproducible results than either the oxalic acid or hydroxylamine hitherto used. After reduction, the mercury is determined by extraction with successive amounts of a $CHCl_3$ soln. of dithizone, added from a burette, the end-point being indicated by a change of colour from orange to green - grey, the precise tint being selected by the operator. A preliminary titration of an aliquot is necessary to locate the approx. position of the end-point, so that, as it is approached, suitably small additions (0·5 ml) of the extracting titrant

may be made. The dithizone soln. is standardised against known amounts of $HgCl_2$ in $KMnO_4$ soln. by the same procedure with the same selected end-point colour, and a calibration graph is constructed. The graph departs slightly from linearity and the procedure is rigorously standardised. Interference by other metals is discussed. A. O. JONES

275. **Colorimetric micro-determination of chloropicrin in air.** R. M. Fournier and M. Person. *Chim. Anal.*, 1957, **39** (7), 263–266.—The two procedures described are based on the reaction between chloropicrin (collected in 2-methoxyethanol) and either dimedone or phloroglucinol in the presence of pyridine and KCN, followed by the determination of the extinction of the coloured product. If dimedone is used, a rose coloration is obtained on heating the soln. at 100° for 5 min.; the sensitivity is 0·4 μg of chloropicrin per ml and one determination can be made in ≃7 min. If phloroglucinol is used, the sensitivity is 2 μg per ml (≡ 2 to 8 mg of chloropicrin per cu. metre of air); the colour develops immediately and a quant. result can be obtained in ≃3 min. This procedure can be used to determine the absorption efficiency and retentive capacity of activated C towards chloropicrin.

W. J. BAKER

276. **Application of cerimetry in water analysis.** H. Lecoq (Lab. Chim. Ind. Minérale, Univ. de Liége, Belgium). *Chim. Anal.*, 1957, **39** (6), 232–234.—For testing the oxidisability of waters, $Ce(SO_4)_2$ has advantages over $KMnO_4$. After acidification with H_2SO_4, the sample is boiled under reflux with $N/8$ or $N/80$ $Ce(SO_4)_2$ for 20 min. After cooling, two drops of a reducing indicator, such as ferroin, are added, and the liquid is titrated with $N/8$ or $N/80$ ferrous ammonium sulphate to a colour change from blue to red. W. J. WRIGHT

277. **Micro-determination of dissolved oxygen in water. I. The nature of the problem.** E. C. Potter (Central Electricity Authority, Boiler Res. Lab., St. John's Wood, London). *J. Appl. Chem.*, 1957, **7** (6), 285–297.—The precautions necessary in this determination are critically reviewed. The latest developments in amperometric apparatus provide a means for designing an analytical method to detect 0·001 p.p.m. of dissolved oxygen, to follow a modification of the Winkler procedure.

II. The design of water-sampling vessels. E. C. Potter. *Ibid.*, 1957, **7** (6), 297–308.—The precision of the methods for the determination of dissolved oxygen at concn. below 0·1 p.p.m. is dependent on the accuracy of the sampling and reaction vessels. A vessel in which errors normally due to the retention of reagent in the bore of the tap are eliminated, and which incorporates high accuracy of measurement of the reagents, is described.

III. Titrimetric determination of iodine in submicrogram amounts. E. C. Potter and J. F. White. *Ibid.*, 1957, **7** (6), 309–317.—Amperometric titration of iodine is a rapid, convenient and accurate method for the completion of the micro-determination of dissolved oxygen. A variant of the classical electrical circuit with all-platinum electrodes is described, but good, reproducible results in excellent agreement with those in which starch indicator is used can be obtained with a simple circuit in which a Doran Universal pH meter is connected across a 10 to 50-megohm resistor joining a platinum and a tungsten electrode. The electrode assembly is conveniently contained in a tube (6 mm in diam.) with the electrode sealed through the lower end. Titrations are carried out in a stream of oxygen-free nitrogen which passes over the electrodes. Excess of $Na_2S_2O_3$ soln. is added to the sample and this is back-titrated with KIO_3 soln. until the first current response indicates the end-point. There is no interference from Fe^{2+}, Fe^{3+} and Cu^{2+} at concn. of 2·5 p.p.m.

IV. Test of Winkler's reaction to below 0·001 p.p.m. of dissolved oxygen. E. C. Potter and J. F. White. *Ibid.*, 1957, **7** (6), 317–328.—Details are given for testing the accuracy of Winkler's method in the concn. range 0·0007 to 0·0544 p.p.m. Special apparatus and procedure for the prep. of oxygen-free nitrogen, oxygen-free water and soln. containing standard amounts of dissolved oxygen are described. The results are considered to be accurate within ±0·0006 p.p.m. D. G. FORBES

278. **Estimation of the total chemical oxygen consumption of water.** T. N. Kleinert and W. Wincor (Zellwolle Lenzing A.-G., Lenzing, Oberösterreich). *Öst. ChemZtg*, 1957, **58** (13–14), 177–179.—The method consists in the wet combustion of the sample in the presence of excess of a strongly acid KIO_3 - H_2SO_4 mixture. The excess of KIO_3 is determined by adding KI and titrating the liberated iodine with $Na_2S_2O_3$ soln. A precaution is the prior removal of carbonate hardness and any H_2S which may be present by boiling the sample with the calculated quantity of H_2SO_4. This is determined by titration of a separate sample, with methyl orange as indicator. The method gave good results for river waters containing sulphite effluents, which gave low results when the usual $K_2Cr_2O_7$ - H_2SO_4 mixture was used. L. S. ADLER

279. **New processes for determination of the potassium permanganate demand in water and sewage.** G. Gad and W. Hoppe. *Gesundheitsing.*, 1956, **77**, 343–345.—The $KMnO_4$ remaining unaltered in the 10-min. test and the MnO_2 formed can be converted into a red - violet manganic pyrophosphate by addition of sodium pyrophosphate and $MnSO_4$. The determination may be either colorimetric or by titration with $FeSO_4$, with diphenylamine as redox indicator.

WATER POLLUTION ABSTR.

280. **The determination of ammonia and total ionic inorganic nitrogen in sea water.** J. P. Riley and P. Sinhaseni. *J. Mar. Biol. Ass. U.K.*, 1957, **36**, 161–168.—In the separation of ammonia from sea water by vacuum distillation (*Anal. Abstr.*, 1954, **1**, 825) special apparatus is required and the process is time-consuming. Micro-diffusion with use of a Cavett flask is as satisfactory, diffusion being about 75% complete after 24 hr. at 70°. The recovered ammonia is then determined by a modification of the phenoxide - hypochlorite method. Nitrate and nitrite in sea water can be determined by reduction to NH_3 with Raney nickel in the presence of EDTA; the ammonia is then separated by micro-diffusion and determined colorimetrically. WATER POLLUTION ABSTR.

281. **Recommended methods for the analysis of trade effluents. Methods for the determination of phenols and sulphide.** The Joint A.B.C.M. - S.A.C. Committee on Methods for the Analysis of Trade Effluents. *Analyst*, 1957, **82**, 518–521.—Two methods are recommended for the determination of phenols. Effluents containing 0·1 to 1 mg of phenols per litre (or adjusted to this concn. by dilution) are treated with $NaHCO_3$, a *p*-aminodimethylaniline reagent (prep. described) and $K_3Fe(CN)_6$

soln. The blue complex is extracted with CCl_4 and its extinction is measured at 600 to 610 mμ (or in an absorptiometer with a suitable red filter) and referred to a calibration graph, or the colour may be matched visually against standards. Cyanides and amines are removed by a preliminary procedure. Samples containing more than 20 mg of phenols per litre are brominated with a $KBrO_3$-KBr mixture in the presence of dil. H_2SO_4 and, after 1 hr., KI is added, the liberated iodine is titrated and the Br absorbed is thus determined. To determine total sulphides, H_2S from the acidified sample is displaced by a stream of inert gas and absorbed in Zn acetate soln. It is again liberated from the ZnS by acidification in the presence of standard iodine soln. and the iodine absorbed in its oxidation is determined by titration. If interfering substances are present, the sulphides are pptd. as ZnS, which is collected and treated as described. A. O. Jones

See also Abstract 286.

Agriculture and Plant Biochemistry

282. **Determination of zinc in plant material without use of organic solvents.** R. K. Jackson and J. G. Brown. *Proc. Amer. Soc. Hort. Sci.*, 1956, **68**, 1–5.—Ashed material containing 5 to 100 μg of Zn is digested in 2 *M* HCl and the extract is passed through a column of Dowex-1 resin. The Zn is eluted with 0·005 *M* HCl and determined colorimetrically in the eluate with zincon reagent. The results agree reasonably with those obtained by dithizone extraction, and there is 95% recovery of added Zn. L. G. G. Warne

283. **Determination of phenolic glycosides and aglycones on paper chromatograms.** J. B. Pridham (Inst. of Paper Chem., Appleton, Wis., U.S.A.). *Anal. Chem.*, 1957, **29** (8), 1167–1169.—The sample soln. is applied to the chromatographic paper as small discrete spots, and after development with a suitable solvent the chromatogram is sprayed with a soln. of diazotised *p*-nitroaniline buffered with Na acetate. The spots are cut from the paper and are eluted with a soln. of KOH in aq. methanol. The absorbance of this soln. is measured at 458 mμ for arbutin, 470 mμ for catechol and 495 mμ for saligenin content, and a linear response is obtained over the range 0 to 100 μg for catechol and arbutin and 0 to 70 μg for saligenin. Errors in all cases are within ±4%. The method was applied to the determination of *p*-hydroxyphenyl-β-gentiobioside in enzymic digests. G. P. Cook

284. **The detection of some naturally occurring flavanone compounds on paper chromatograms.** E. Eigen, M. Blitz and E. Gunsberg (U.S. Vitamin Corp., Yonkers, N.Y., U.S.A.). *Arch. Biochem. Biophys.*, 1957, **68** (2), 501–502.—The reagent is freshly prepared as required by dissolving sodium borohydride (0·1 g) in the min. of water and diluting to 10 ml with *iso*propyl alcohol. Paper chromatograms are sprayed with the reagent and then exposed to the vapour of conc. HCl for 15 to 30 sec. Flavanones (< 2 μg) give a purple to magenta colour; flavones and flavanols do not react. W. H. C. Shaw

285. **Improvements in colorimetric determinations. III. Comparison of colorimetric and electrometric methods for the determination of soil reactions.** K. L. Khanna, P. B. Bhattacharya and K. Prasad (Central Sugarcane Res. Sta., Pusa, Bihar). *Proc. Indian Acad. Sci., A*, 1957, **45** (2), 89–90.—The pH values of 300 soil samples (non-calcareous soils with low soluble-salt content) were determined by the electrometric and colorimetric methods. The results were in close agreement. I. Jones

286. **Comparison of flame-photometric and chemical methods for determining sodium and potassium in soil, plant material, water and serum.** M. Puffeles and N. E. Nessim (Lab. for Chem. Anal., Min. of Agric., Tel-Aviv, Israel). *Analyst*, 1957, **82**, 467–474.—The work described was carried out with an E.E.L. single-cell flame photometer. Expt. to determine the effect of Ca on the determination of Na (10 p.p.m.) showed an error ranging from +14% with 50 p.p.m. to +52% with 500 p.p.m. of Ca. In the determination of K, the error due to Ca is negligibly small below 300 p.p.m. of Ca. Similar expt. on the determination of Na and K in the presence of PO_4^{3-} confirmed the report of Collin *et al.* (*Analyst*, 1952, **77**, 430) that these ions cause negative errors in the determination of both Na and K, the effect on K being by far the greater. Determinations were made of the Na and K contents of soil extracts, plant ash, natural waters and blood sera, both by the flame photometer and by chemical methods, the Na gravimetrically as sodium magnesium uranyl acetate and the K volumetrically as cobaltinitrite. The results showed that, when the flame-photometric results are corrected as described and the chemical results are corrected for low recovery (97·6 ± 0·3%), the agreement was generally acceptable. A. O. Jones

287. **Rapid method for the determination of chromium in soils for use in geochemical prospecting.** G. A. Wood and R. E. Stanton (Dept. of Geology, Imperial Coll. of Science and Technol., London). *Trans. Inst. Min. Metall.*, 1957, **66** (7), 331–340.—Procedures are described whereby Cr, in the concn. range of 250 to 30,000 p.p.m., can be determined by visual comparison of alkaline chromate soln. with standards, and, in concn. down to 5 p.p.m., by colorimetric reaction with diphenylcarbazide. The accuracy of these procedures is within ±25%, which is satisfactory for normal geochemical prospecting purposes. S.C.I. Abstr.

288. **Analyses of a chromatographic fraction of organic extracts of soils.** W. G. Meinschein and G. S. Kenny (Magnolia Petroleum Co., Dallas, Texas, U.S.A.). *Anal. Chem.*, 1957, **29** (8), 1153–1161.—The benzene eluates of the soil extracts from silica gel chromatographic columns contain a high concn. of waxes of the types appearing in beeswax. Qual. and quant. analysis of the soil waxes were accomplished by chromatographic, i.r. and mass-spectrometric methods in conjunction with hydrogenation studies. The principal constituents of the waxes are normal aliphatic acids, normal primary aliphatic alcohols and sterols. All the extracts contained pentacyclic and hexacyclic compounds, which may be triterpenes. G. P. Cook

289. **Estimation of substituted thio-oxazolidones in rape-seed meals.** L. R. Wetter (Prairie Regional Lab., Saskatoon, Saskatchewan, Canada). *Canad. J. Biochem. Physiol.*, 1957, **35** (5), 293–297.—Methods for the estimation of *iso*thiocyanate (**I**) (*cf. Anal. Abstr.*, 1956, **3**, 1151) and thio-oxazolidone (**II**) (*cf.* Astwood *et al.*, *J. Biol. Chem.*, 1949, **181**, 121) are modified in order to determine both constituents in one portion of rape-seed meal. A

soln. of myrosinase (0·5%) in saline (0·9%) replaced white mustard powder to effect the splitting of glycosides in the sample. *Procedure*—Estimate **I** (*loc. cit.*) in a portion of the sample meal. Filter off the residue from the distillation and adjust an aliquot of the filtrate to pH 10·5 with *N* NaOH to prevent interference by citrates. Extract the soln. with diethyl ether (2 × 10 vol.) and discard the aq. layer. Measure the absorption of the mixed ether extracts at 230, 248 and 266 mμ with a Beckman DU spectrophotometer. Estimate **II** by relating the corrected absorption at 248 mμ [(absorption at 248 mμ) − (average absorption at 230 and 266 mμ)] to a standard curve.

D. G. FORBES

290. **Determination of added cobalt in mixed feeding salts.** W. A. C. Campen and H. Dumoulin (Rijkslandbouwproefstation, Maastricht, Holland). *Chem. Weekbl.*, 1957, **53** (29), 398–400.—Improvements in an earlier method (*cf.* Campen and Geerling, *Brit. Abstr. C*, 1952, 483) enable only the rapidly sol. cobalt salt, about 0·004%, to be determined within 15 min. The ground sample (2 to 5 g) is mixed with acetate - thiourea buffer (pH 4) (5 ml), 60% NH_4SCN soln. (45 ml) and amyl alcohol - ether (35:65) (40 ml). The blue extract is made up to 50 ml and its extinction measured at 620 mμ. This method is not applicable to seaweed or bran.

P. RENTENAAR

291. **Colorimetric determination of *p*-chlorobenzyl *p*-chlorophenyl sulphide [chlorbenside] as a spray residue.** H. J. Hardon, H. Brunink and E. W. van der Pol (Keuringsdienst van Waren, Amsterdam, Holland). *J. Sci. Food Agric.*, 1957, **8** (6), 368–370.—The use of CrO_3 rather than H_2O_2 as the oxidising agent for converting the chlorbenside into the corresponding sulphone (*cf.* Higgons and Kilbey, *Anal. Abstr.*, 1956, **3**, 589) eliminates interference by plant waxes, and the coloured complex produced by treatment of the sulphone with *m*-dinitrobenzene reagent is not given by DDT. *Procedure*—Peelings from the fruit are covered with light petroleum, set aside overnight, then filtered. The solvent is distilled off, and the gelatinous residue is boiled with CrO_3 soln. (5% in glacial acetic acid). The resulting soln. is extracted with a mixture (1:1) of ether and light petroleum, and, after filtration of the extract through anhyd. Na_2SO_4, the solvent is distilled off. Ethanol, 2% ethanolic *m*-dinitrobenzene and 2·5 *N* KOH are mixed with the residue and maintained at 25° for 60 min., and the product is cooled in a refrigerator for 10 min. to stabilise the colour before measuring the extinction at 510 mμ. The recovery is >90%.

D. G. FORBES

292. **Colorimetric analysis of the isomers of tetrachloronitrobenzene and evaluation of deposits on the soil obtained from smokes of the 2:3:5:6-isomer (tecnozene).** D. J. Higgons and A. Toms (Boots Pure Drug Co., Nottingham, England). *J. Sci. Food Agric.*, 1957, **8** (6), 309–313.—The estimation of 2:3:5:6-tetrachloronitrobenzene (**I**) in the presence of the 2:3:4:6- and the 2:3:4:5-isomers is described. *Procedure*—Pipette aliquots of a standard soln., containing 5 to 100 μg of **I** dissolved in light petroleum (boiling range 40° to 60°), into separate flasks, and evaporate off the solvent. To each flask add 0·1 *N* Na methoxide in methanol (2 ml), and reflux gently for 2 hr. Cool and transfer the liquids to 50-ml calibrated flasks and add water to 30 ml. To each flask add sulphanilic acid soln. (0·6 g is dissolved in hot H_2O, 20 ml of conc. HCl is added to the cool soln., and the mixture is diluted to 100 ml) (1 ml), and set aside for 10 min. Add 1-naphthylamine hydrochloride soln. (0·6 g of 1-naphthylamine hydrochloride in water containing 1 ml of conc. HCl, diluted to 100 ml) (1 ml), followed by Na acetate soln. (20%) (1 ml). Dilute the liquids to 50 ml and mix well. Measure the intensity of the resulting red colours in a 4-cm cell by means of a Spekker absorptiometer, with Ilford 604 and heat-resisting filters and a water-to-water setting of 1·0. Deposits on filter-papers which had been exposed to smokes of **I** were extracted with light petroleum (3 × 15 ml), the extracts were made up to 50 ml, and 1 ml was treated as for the standards. A 2-hr. reflux period ensures reproducible results, and recovery is >90%, but there is a little interference from the 2:3:4:6-isomer.

D. G. FORBES

See also Abstracts 27, 139, 231.

5.—GENERAL TECHNIQUE AND LABORATORY APPARATUS

General

293. **Sensitive quartz beam microbalance.** A. W. Czanderna and J. M. Honig (Dept. Chem., Purdue Univ., Lafayette, Ind., U.S.A.). *Anal. Chem.*, 1957, **29** (8), 1206–1210.—A rugged, sensitive microbalance which can be constructed, maintained and operated with normal experimental skill is described. It operates on the principle of a normal gas density balance and is capable of detecting mass changes to a precision of 5×10^{-8} g and to an accuracy of $\pm 10^{-7}$ g. With proper temp. control it is stable over long intervals of time.

K. A. PROCTOR

294. **Automatically recording vacuum balance.** M. I. Pope (Washington Singer Lab., Univ. Exeter, England). *J. Sci. Instrum.*, 1957, **34** (6), 229–232.—The apparatus comprises an electromagnetic sorption balance having a split plane mirror fixed to the beam so that a beam of light reflected from it falls equally on to two photocells when in the null position. The maintenance of the null position is effected by automatically regulating the current in a solenoid surrounding the balance magnet. The instrument is capable of recording changes in weight of a solid at temp. up to 1000° to within ±0·2 mg, with a total range of 5 g.

G. SKIRROW

295. **Effect of temperature on the precision and performance of a microchemical balance.** D. E. Hull (California Res. Corp., Richmond, U.S.A.). *Anal. Chem.*, 1957, **29** (8), 1202–1204.—By maintaining the ambient temp. at 30°, a precision of 1·0 μg in rapid, routine weighing was attained on an Ainsworth Type FDJ microchemical balance. A larger standard deviation was found at 25° and is attributed to thermal disturbances caused by heat from the operator's hands and face. A rhythmic procedure is proposed for weighing at room temp. in order to minimise temp. effects on the precision of weighing.

K. A. PROCTOR

296. **Autodifferential dilatometer.** W. L. De Keyser (Univ. de Bruxelles, Belgium). *Chim. Anal.*, 1957, **39** (6), 229–231.—The apparatus embodies the principle of the differential thermobalance. It permits the measurement of differences in length of two identical samples of which the temp. during heating varies by a constant factor. Differentials of the dilatometric curve are thus obtained. The apparatus is particularly applicable to the study of ceramic materials. W. J. WRIGHT

297. **Selective measurement of traces of gas with automatic analytical apparatus.** H. Fuhrmann and H. Galster (Bran & Lübbe, Hamburg, Germany). *Chem.-Ing.-Tech.*, 1957, **29**, 256–258.—The apparatus described (the Chromoflux) provides a highly sensitive means for the continuous and automatic measurement of gases. The gas is absorbed in a glass spiral by counter-current action with a suitable solvent. The absorbent then drops into a glass cell where its optical extinction is measured photometrically. J. H. WATON

298. **Stationary thermobarometer for rotary Warburg apparatus.** L. Ferrara (International Minerals and Chemical Corp., Chicago, Ill., U.S.A.). *Anal. Chem.*, 1957, **29** (8), 1239.—A modification of the rotary Warburg apparatus in order to increase the precision and ease of operation is described. By fitting a stationary thermobarometer, a shorter time-lapse occurs between readings of the thermobarometer and each manometer, and this minimises errors due to any intermittent and rapid changes in the temp. of the bath and pressure of the surrounding atmosphere. K. A. PROCTOR

299. **Universal titration console.** E. L. Duggan and V. L. Stevens (School of Medicine, Univ. of California, Berkeley, U.S.A.). *Anal. Chem.*, 1957, **29** (7), 1076–1079.—An apparatus which enables titrations to be carried out under definite conditions of mixing of the reagents, rate of titrant delivery and temperature is described. Results, which are recorded, are obtained potentiometrically.
D. A. PANTONY

300. **A simple burette for titanimetric titrations.** O. Vacek and O. Leminger (Org. Lab., Spolek pro Chem. a Hutní výrobu, Ústí nad Labem, Czechoslovakia). *Chem. Listy*, 1957, **51** (6), 1209–1210.—The new apparatus described is suitable for titrations with $TiCl_3$, $CrCl_2$ and $SnCl_2$; the soln. to be determined is protected with a layer of a liquid. A magnetic stirrer must be used. J. ZÝKA

301. **An improved all-glass extractor.** H. E. Hadd and W. H. Perloff (Temple Univ. School of Med., Philadelphia, Pa., U.S.A.). *J. Clin. Endocrin. & Metabolism*, 1957, **17** (5), 673–675.—An automatic liquid - liquid extractor, working at room temp., that can be used for extracting urine, blood and tissue homogenates with solvents heavier or lighter than water, is described.
H. F. W. KIRKPATRICK

302. **Analytical distillation laboratory design for efficiency and versatility.** C. B. Kincannon and M. O. Baker (Manuf. Res. Lab., Shell Oil Co., Houston, Tex., U.S.A.). *Anal. Chem.*, 1957, **29** (8), 1189–1193.—The laboratory contains 18 column stalls to allow fractionating columns to be interchanged, and reduces man- and column-hours per distillation by about 25% and 85%, respectively. Failures average about 1% and repeatability is improved by about 25%. Selected operating pressures are maintained automatically, and constant boil-up rates are obtained by semi-automatic control. Control panels allow for the rapid selection of reflux ratios and for reading vapour temp.
K. A. PROCTOR

303. **Automatic receiver for continuous vacuum distillation.** W. J. Humphlett (Eastman Kodak Co., Rochester, N.Y., U.S.A.). *Anal. Chem.*, 1957, **29** (8), 1241–1242.—The receiver described can be used to collect the distillate automatically without disturbing the pressure or ebullition in the distilling apparatus. K. A. PROCTOR

304. **Bulb-tube assembly for vacuum distillation.** A. W. Schrecker (Nat. Cancer Inst., Bethesda, Md., U.S.A.). *Anal. Chem.*, 1957, **29** (7), 1113–1114.—A simple bulb-type apparatus for vacuum distillation or sublimation is described. D. A. PANTONY

305. **Improvised traps for vacuum lines.** M. Bloom (Hughes Aircraft Co., Culver City, California). *J. Chem. Educ.*, 1957, **34**, 173.—The heating chamber portion of an Abderhalden drying apparatus is mounted with its main axis vertical and the sample chamber filled with a suitable refrigerant. The ground glass joints normally used for attaching the boiling flask and reflux condenser form part of the vacuum line. S.C.I. ABSTR.

306. [**Apparatus for use in connection with**] **the colorimetric determination of fluorine.** A. A. Guntz and M. Arène. *Chim. Anal.*, 1957, **39** (7), 260–262.—The usual entrainment - distillation apparatus for the extraction of fluorine as H_2SiF_6 by the Willard - Winter method is simplified by inserting the extractor (containing the sample) in a conical boiling flask containing $\simeq$670 ml of 60% H_2SO_4, which ensures a max. b.p. of 157°. If the outlet of the extractor includes a steam trap, the condensate should contain only $\simeq$0·001% of H_2SO_4, which does not affect the subsequent colorimetric procedure. Control is automatic and no thermometer is needed; either Pyrex glass or boron-free glass can be used for all the parts. If the extraction is made with $HClO_4$, the max. necessary temp. is 135°. W. J. BAKER

307. **Technique for vacuum filtration.** J. R. Brown (Veterans Admin. Res. Hospital, Chicago, Ill., U.S.A.). *J. Chem. Educ.*, 1957, **34**, 165.—The use of moderately rigid rubber with smooth surfaces to make a tight connection between bell-jar and receiver enables filtration to be made directly into a variety of receivers, obviating the need to transfer from a suction flask or to rinse down a filtering adapter. S.C.I. ABSTR.

308. **Two techniques for paper chromatography.** D. I. French and M. R. Gibson (School of Pharmacy, State Coll. of Wash., Pullman, U.S.A.). *Anal. Chem.*, 1957, **29** (8), 1166–1167.—Two apparatuses are described. The first, for extraction, is used to extract alkaloids from crude solanaceous drugs. The entire extract is placed on a small spot of a paper strip and is developed in the normal manner with an appropriate solvent. The alkaloids are detected with Dragendorff reagent, the alkaloid sections are cut and are placed in the second apparatus, for elution, which was found to be 99·6 $\pm$ 0·4% efficient. The eluates are assayed spectrophotometrically by the Vitali morin reaction.
G. P. COOK

309. **New quantitative chromatographic filter-paper method utilising a battery of microchromatograms with bridges.** T. Braun. *Rev. Chim., Bucharest*, 1957, **8** (2), 127–129.—A rectangular piece of filter-paper is specially cut so that six narrow strips are formed, bridged together at the bottom. After irrigation and development of the substance being chromatographed, streaks, and not the usual spots, are obtained, the length of which are in log. ratio to the concn. of materials present. For estimations, the first five strips are treated with an equal vol. of increasing standard concn. of the pure substance to be estimated, while the sixth strip is treated with an identical vol. of the soln. to be analysed. The amount present is extrapolated from the graph obtained from the first five strips. The error in the estimation of hydrazine, ammonia, urea, Mo and U is claimed to be ±8%. H. SHER

310. **Glass strip collector for column chromatography.** R. A. Dewar and D. Gunew (Central Res. Lab., ICIANZ, Ltd., Melbourne, Australia). *Nature*, 1957, **179**, 1245.—The device described presents a visible result of an analysis in a manner similar to one-dimensional paper chromatography, but without the need for specific development reagents. Fractions can be isolated and estimated quant. They are collected on a serrated glass strip which rests on a carriage moving at constant speed beneath the drip point. The solvent is evaporated at the drip point by heat from an infra-red lamp. Black glass is used for colourless crystalline substances. Oily fractions are developed by dusting with talc. L. S. ADLER

311. **A practical aid in paper chromatography.** H.-J. Mierau (Inst. of Hygiene, Hamburg, Germany). *Dtsch. ApothZtg*, 1957, **97** (9), 185.—Good chromatographic separations from small quantities of material can be achieved with a simple apparatus, which comprises two watch-glasses placed together to form a cell containing the solvent. The upper glass is perforated, and on its upper side lies the paper containing the spot. The solvent is supplied to the paper by means of a paper wick, and rapid spreading of the solvent front is prevented by a jet of warm air directed on to the paper from above. The paper is held in position by a lead ring. With such an apparatus it is possible to operate for 15 min. and produce a circular spot of 0·6 cm in diam. from not more than 0·5 ml.
G. R. WHALLEY

312. **Application of combined paper ionophoresis and paper chromatography.** C. G. Honegger (Forschungslab. Psychiatrischen Univ.-Klinik, Basel). *Helv. Chim. Acta*, 1957, **40** (3), 846–851.—An improved and simplified elution technique is described for a separation on two papers. A new arrangement for the separation of difficultly elutable mixtures with two-dimensional ionophoresis - chromatography on one paper is described and applied to the aq. HCl extract of a carcinoid metastasis. M. DAVIS

313. **Rapid, precise micro vapour pressure method.** A. Y. Mottlau (Products Res. Div., Esso Research & Engineering Co., Linden, N.J., U.S.A.). *Anal. Chem.*, 1957, **29** (8), 1196–1202.—The measuring device is based on the evacuable bulb principle with the Harris sample-introduction system of a mercury-sealed orifice as used for introducing gas and liquid samples into the inlet systems of mass spectrometers. This method requires a sample charge of only 1 ml and is more precise and faster than the Reid method ("A.S.T.M. Standards on Petroleum Products and Lubricants," Method D 323–52, 1954). The apparatus is designed to give results that correlate with those obtained by the Reid method.
K. A. PROCTOR

See also Abstracts 105, 173, 336.

Optical

314. **Optical and instrumental limitations to the accuracy of "oximetry."** R. H. Kay and R. V. Coxon (Univ. Lab. Physiol., Oxford). *J. Sci. Instrum.*, 1957, **34** (6), 233–236.—An "oximeter" is a miniature filter photometer for measuring the oxygen saturation of haemoglobin by comparing the light transmission in two separate spectral regions. With an instrument based on the design of Stott (*J. Sci. Instrum.*, 1953, **30**, 120), it was found that a large contribution to the total light extinction derived from scattering effects in whole blood. This factor, together with instrumental deficiencies, limits the accuracy of the method on whole blood in glass absorption cells to ±8% oxygen saturation. N. E.

315. **Improvements relating to the flame-photometric testing of flammable gases and apparatus therefor.** Stamicarbon N.V. Brit. Pat. 779,212; date appl. 19.4.55.—An apparatus is described by which the luminosity of a flame of burning gas is compared with the luminosity of a flame from which one or more specific components contributing to the luminosity have been removed. The method is particularly applied to the determination of "benzol" in coke-oven gas. N. E.

316. **Improvements in and relating to cell holders for spectrophotometers.** National Research Council. Brit. Pat. 779,309; date appl. 28.6.55.—The invention describes an arrangement for temperature control of a cell holder for use with a spectrophotometer. N. E.

317. **Method of monitoring spectroscopic sources.** F. R. Bryan and E. F. Runge (Ford Motor Co., Dearborn, Mich., U.S.A.). *Appl. Spectroscopy*, 1957, **11** (2), 64–67.—A means of controlling spark excitation in order to improve the accuracy of spectrochemical analyses is described; it is applicable to air-interrupted sources regardless of the type of analysis being performed. The control system consists of a photocell pick-up triggering an electronic scaling circuit. Illumination from the spark discharge produces the photocell signal. Individual discharges are counted to provide an indication of the exact number of spark discharges per unit time.
K. A. PROCTOR

318. **Photo-electric spectrometer for the examination of steels.** H. Diebel and W. Hanle. *Arch. Eisenhüttenw.*, 1957, **28** (3), 127–132.—In this Report No. 237 of the Chemikerausschusses des Vereins Deutscher Eisenhüttenleute, the construction of a photo-electric spectrometer, its operation and its installation in a steel works are described. The instrument comprises a spark source, a 2-m grating, photomultipliers, integrators and measuring apparatus. The choice of a grating instead of a prism is discussed. The switching arrangements and associated equipment are described. The apparatus is used for the determination of Si, Mn, Cr, Ni, Mo and Cu. G. BURGER

319. **Development possibilities of photo-electric emission-spectrum analysis.** H. Krempl and G. Scheibe. *Arch. Eisenhüttenw.*, 1957, **28** (3), 135–139.—In this Report No. 239 of the Chemikerausschusses des Vereins Deutscher Eisenhüttenleute, present difficulties and limitations are reviewed. Promising lines of research include study of line intensities in different atmospheres, the mechanics of emission from single sparks, and the design of spark generators and the combinations of parameters required, particularly for the more difficult elements. A prism arrangement is described which, because of its high dispersion, is particularly suitable for steel analysis. An automatic instrument has been developed in which photomultipliers, integrators and a recorder is used. The use of spectral charts in adjusting the instrument is discussed.
G. BURGER

320. **Water-cooled mercury arc lamp for the excitation of Raman spectra.** L. A. Woodward and D. N. Waters (Inorg. Chem. Lab., Oxford). *J. Sci. Instrum.*, 1957, **34** (6), 222–224.—The construction and operating characteristics of the Pyrex-glass Toronto-type mercury vapour lamp are described. Provision is made for the cooling of the electrodes and the whole length of the arc (185 cm), which is in the form of an inverted U. For a given current the light output is slightly lower when the lamp is running with full cooling than when it is used as an ordinary Toronto arc, but full cooling permits the use of higher currents, thus giving greater light output.
G. SKIRROW

321. **Wavelength marker for spectrophotometers incorporating a strip chart recorder.** K. M. M. Kruse (Council for Sci. Ind. Res., Pretoria, S. Africa). *Anal. Chem.*, 1957, **29** (8), 1240–1241.—The device described enables the wavelength scale to be directly recorded together with the spectrum on a Beckman Model DK-1 spectrophotometer. This makes the exactness of the wavelength scale independent of contraction or expansion of the chart paper due to changes in humidity and temp., and facilitates the use of uncalibrated wavelength charts. On one instrument calibrated with the lines of a mercury arc between 194 and 2320 mμ, the reproducibility of wavelength readings was better than 0·6 mμ and 0·02 mμ in the 2000-mμ and 200-mμ regions, respectively.
K. A. PROCTOR

322. **Microlitre absorption cell and its adaptation to the Beckman Model DU spectrophotometer.** D. Glick and B. W. Grunbaum (Univ. Minnesota, Minneapolis, U.S.A.). *Anal. Chem.*, 1957, **29** (8), 1243–1244.—With the cell described, the extinctions of 5-μl aliquots of three concn. of sulphobromophthalein, each in a different cell, were measured with a max. coeff. of variation of $\pm 1\%$ (10 replicates).
K. A. PROCTOR

323. **Infra-red spectrophotometric analysis of fractional milligram quantities of solids.** J. J. Kirkland (E.I. du Pont de Nemours & Co., Inc., Wilmington, Del., U.S.A.). *Anal. Chem.*, 1957, **29** (8), 1127–1129.—An evacuable, rectangular die for pressing small potassium bromide pellets is described. The die serves as the pellet holder in a Perkin - Elmer Model 21 i.r. spectrophotometer. In this way very small quantities of sample can be examined qual. and quant. under standard conditions without auxiliary beam-condensing equipment.
K. A. PROCTOR

324. **General procedure for setting up infra-red differential analyses of multicomponent mixtures.** J. A. Perry and G. H. Bain [Standard Oil Co. (Indiana), Whiting, Ind., U.S.A.]. *Anal. Chem.*, 1957, **29** (8), 1123–1127.—The precision of i.r. analyses of multicomponent mixtures can be greatly improved by using differential spectrophotometry with wire screens (*e.g.*, sections of 16-mesh wire gauze) as standards of absorbance. A sequence of steps for straightforward calibration is illustrated in detail by the analysis of a 6-component C_4 hydrocarbon gas.
K. A. PROCTOR

325. **Agitating system for gases in infra-red absorption cell.** A. L. Olsen and H. W. Kruse (U.S. Naval Ordnance Test Sta., China Lake, Calif., U.S.A.). *Anal. Chem.*, 1957, **29** (8), 1242.—The cell described is fitted with a 300 r.p.m. system and is designed for use in the Perkin - Elmer Model 21 spectrophotometer.
K. A. PROCTOR

326. **Adaptation of a spectrometer as an absolute Pulfrich refractometer.** W. Primak (Argonne Nat. Lab., Lemont, Ill., U.S.A.). *Anal. Chem.*, 1957, **29** (8), 1237–1239.—Details are given of the arrangement of the spectrometer for the determination of prism constants and the determination of the refractive index of a small plate.
K. A. PROCTOR

Thermal

327. **Melting-point determination to 0·1 of a degree. II.** K. Rast (Farbenfabriken Bayer A.-G., Leverkusen, Germany). *Chem.-Ing.-Tech.*, 1957, **29**, 277–279.—The two-bath apparatus previously described (*Ibid.*, 1955, **27**, 523, 626) for determining m.p. in the range 100° to 285° to an accuracy of 0·1° is modified for the determination of m.p. up to 350°. The liquid used in the baths for the higher temp. range is a melt of $NaNO_3$ - KNO_3 - $RbNO_3$ (4:5:10). The outer bath is dispensed with and is replaced by an iron sphere; the bath liquid, contained in a glass tube, is inserted into a hole cut in the sphere. The list of substances whose m.p. constitute the fixed-point scale is given for the full range 100° to 350°.
J. H. WATON

328. **Apparatus giving thermogravimetric and differential thermal curves simultaneously from one sample.** D. A. Powell (Div. of Bldg. Res., C.S.I.R.O., Melbourne). *J. Sci. Instrum.*, 1957, **34** (6), 225–227.—Details are given of modifications to a photographic recording Chevenard thermal balance.
G. SKIRROW

Electrical

329. **Improvements in or relating to gas detection method and apparatus.** Mine Safety Appliances Co. Brit. Pat. 776,770; date appl. 11.2.54. U.S.A., date appl. 26.2.53.—A method for the electrochemical detection of an oxidising gas utilises a polarised electric detector cell having a zinc anode and a carbon cathode adapted to be depolarised by the gas, the half-cell e.m.f. of the carbon electrode being equal to zero in the absence of the oxidising gas. A known concn. of the oxidising gas is admitted to the carbon electrode and the total electric power output (product of the current and the e.m.f.) produced by the cell is measured. A gas

mixture containing the oxidising gas at an unknown concn. is admitted to the electrode, and the total electric power output produced by the cell is again measured. The unknown gas concn. is determined from the proportionality between the total electric power output and the gas concn. J. M. JACOBS

330. **Electrochemical detection of oxidising gases.** Mine Safety Appliances Co. Brit. Pat. 776,771; date appl. 11.2.54. U.S.A., date appl. 26.2.53.—A galvanic cell for the determination of the concn. of oxygen in a gas mixture containing other oxidising gases comprises a zinc anode, a carbon cathode, and an electrolyte, *e.g.*, a dil. soln. (2 to 5%) of NH_4Cl acidified with HCl or H_2SO_4 to a pH of $<2{\cdot}8$ (3·0), at which point no development of H at the anode is observed. The area of interaction between the polarised electrode and the electrolyte is smaller than the active area of the other electrode by at least an order of magnitude, and the smaller electrode area is in inverse proportion to the max. concn. of the oxidising component to be tested with a given electric meter. J. M. JACOBS

331. **Zone electrophoresis on sponge rubber.** H. K. Mitchell and L. A. Herzenberg (California Inst. Technol., Pasadena, U.S.A.). *Anal. Chem.*, 1957, **29** (8), 1229.—Foam-rubber sponge is a convenient and useful supporting medium for zone electrophoresis of proteins and other substances. Samples can be recovered afterwards simply by squeezing the sponges. K. A. PROCTOR

332. **Improvements in or relating to polarographs.** Cambridge Instrument Co., Ltd., and G. Jessop. Brit. Pat. 776,543; date appl. 1.7.53.—Reduction of the effect of series resistance in polarograph apparatus is obtained by supplementing the electrode voltage by a further alternating voltage, the amplitude of which is proportional to the electrode current, and which is of the same phase as the electrode current. Thus, *e.g.*, in the arrangement described in B.P. 640,768, the electrode current is amplified by a suitable amplifier, at an output of which, before rectification, a voltage which is of the same phase as, and proportional in amplitude to, the electrode current is obtained. A predetermined fraction of this voltage is superimposed on the voltage applied to the electrode. If this fraction is properly adjusted according to the value of the series resistance, the resulting total electrode current is of the same magnitude and phase as it would be if there were no series resistance. J. M. JACOBS

333. **Calibration of an integrating motor for coulometric titrations.** N. H. Furman and A. J. Fenton, jun. (Frick Chem. Lab., Univ., Princeton, N.J., U.S.A.). *Anal. Chem.*, 1957, **29** (8), 1213–1214.—Extensive calibration has shown that an empirical relationship which is linear over a range of current levels and resistances exists between the motor calibration factor and the value of the series resistor. By using this relationship, motor factors can be calculated with an accuracy of 1 to 2 parts per thousand. K. A. PROCTOR

334. **A solid state paramagnetic resonance spectrometer.** P. M. Llewellyn (Clarendon Lab., Univ. Oxford). *J. Sci. Instrum.*, 1957, **34** (6), 236–239.—A spectrometer is described which is a development of that described by Bleaney and Stevens (*Rep. Progr. Phys.*, 1953, **16**, 108) and is used in the frequency range 9000 to 10,000 Mc/s at the fixed temp. of boiling refrigerants, the lowest temp. being the triple point of hydrogen (14° K). A high sensitivity is achieved by modulating the magnetic field at 115 kc/s. The sensitivity is such that 10^{13} atoms of ^{56}Co have been detected at 20° K, and it is estimated that 2×10^{-11} mole of the free radical diphenyltrinitrophenylhydrazyl would be detectable at 290° K. N. E.

335. **Mass spectrometry applied to clinical practice and research.** K. T. Fowler and P. Hugh-Jones (Postgrad. Med. School, London). *Brit. Med. J.*, 1957, 1205–1211.—An instrument designed specifically for the study of human respiratory function is described. Four gases, in the mol.-wt. range 18 to 80, can be simultaneously and continuously determined throughout the respiratory cycle by using only 0·1% of the patient's exhaled breath. The response time is less than 0·1 sec., changes in concn. of 1% can be detected, and the instrument is transportable. H. F. W. KIRKPATRICK

336. **Oscillographic mass-spectrometric monitoring of gas chromatography.** J. C. Holmes and F. A. Morrell (Philip Morris Inc., Richmond, Virginia, U.S.A.). *Appl. Spectroscopy*, 1957, **11** (2), 86–87.—A method is described for continuously monitoring the eluate from a gas-chromatographic column with a mass spectrometer and an oscilloscope. Any sixteen-mass-unit portion of a spectrum can be displayed and the smallest change in the pattern immediately detected. The scan rate can be altered to decrease the time required for the display or to present a larger portion of the spectrum. The method is applicable to the study of reaction rate, thermal cracking, etc., and the presence of intermediate compounds formed during a reaction can be detected quickly. A commercially available mass spectrometer can be easily adapted for this purpose. K. A. PROCTOR

337. **Neutron moisture-meter for concrete.** J. Pawliw and J. W. T. Spinks (The Univ., Saskatoon, Saskatchewan, Canada). *Canad. J. Technol.*, 1957, **34** (8), 503–513.—A portable instrument for determining rapidly the total content of H_2O in a plain or lightly reinforced concrete member, without destruction, is described. Fast neutrons from a Ra-Be source (10 or 50 mC) are converted into slow neutrons by the H_2O mol. and are then determined with a BF_3 counter (2 or 4 in. long). The meter is calibrated against concrete cylinders of known H_2O content; its sensitivity is less than that of the similar soil-moisture meter because it is placed against the concrete surface. W. J. BAKER

ERRATA.—October (1957) issue, abstract 3240.
Line 7, *for* "HNO_3" *read* "nitrous fumes";
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ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use of an abbreviation or symbol the word is printed in full.

alternating current	a.c.
ampere	amp.
Ångström unit	Å
anhydrous	anhyd.
approximate, -ly	approx.
aqueous	aq.
atmospher-e, -ic	atm.
boiling-point	b.p.
British thermal unit	B.Th.U.
calorie (large)	kg-cal.
calorie (small)	g-cal.
centimetre	cm
coefficient	coeff.
concentrated	conc.
concentration	concn.
constant	const.
corrected	(corr.)
crystalline / crystallised	cryst.
cubic	cu.
current density	c.d.
cycles per second	c/s
density	ρ
density, relative	d or wt. per ml
dilute	dil.
direct current	d.c.
distilled	dist.
ethylenediaminetetra-acetic acid	EDTA
electromotive force	e.m.f.
equivalent	equiv.
gram	g
gram-molecule	mole
half-wave potential	$E_{\frac{1}{2}}$
hour	hr.
hydrogen ion exponent	pH
infra-red	i.r.
insoluble	insol.
international unit	i.u.
kilogram	kg
kilovolt	kV
kilowatt	kW
liquid	liq.
maxim-um, -a	max.
melting-point	m.p.
microgram	μg (not γ)
microlitre	μl
micromole	μmole
micron	μ
milliampere	mA
milli-equivalent	milli-equiv.
milligram	mg
millilitre	ml
millimetre	mm
millimicrogram	mμg
millimolar	mM
millivolt	mV
minute (time)	min.
molar (concentration)	M
molecul-e, -ar	mol.
normal (concentration)	N
optical rotation	α_λ^t
ounce	oz
parts per million	p.p.m.
per cent.	%
per cent. (vol. in vol.)	%(v/v)
per cent. (wt. in vol.)	%(w/v)
per cent. (wt. in wt.)	%(w/w)
potential difference	p.d.
precipitate (as a noun)	ppt.
precipitated	pptd.
precipitating	pptg.
precipitation	pptn.
preparation	prep.
qualitative, -ly	qual.
quantitative, -ly	quant.
recrystallised	recryst.
refractive index	n_λ^t
relative band speed	R_v
relative humidity	r.h.
revolutions per minute	r.p.m.
saponification value	sap. val.
saturated calomel electrode	S.C.E.
second (time)	sec.
soluble	sol.
solution	soln.
specific gravity	sp. gr.
specific rotation	$[\alpha]_\lambda^t$
square centimetre	sq. cm
standard temp. and pressure	s.t.p.
temperature	temp.
ultra-violet	u.v.
vapour density	v.d.
vapour pressure	v.p.
volt	V
volume	vol.
watt	W
wavelength	λ
weight	wt.

In addition, the following symbols may be used in conjunction with numerical values or in mathematical expressions—

greater than	$>$
not greater than	$\ngtr$
is proportional to	$\propto$
less than	$<$
not less than	$\nless$
of the order of, approximately	$\simeq$

The principal Pharmacopoeias are denoted by B.P., U.S.P. or D.A.B., together with the identifying roman numeral or year.

Valency states are represented by a superscript roman numeral, *e.g.*, Fe^{II}, Mo^{V}. Substances in the ionic state are represented by Na^+, Fe^{2+}, Fe^{3+}, etc., for cations and by Cl^-, SO_4^{2-}, PO_4^{3-}, etc., for anions.

mixture containing the oxidising gas at an unknown concn. is admitted to the electrode, and the total electric power output produced by the cell is again measured. The unknown gas concn. is determined from the proportionality between the total electric power output and the gas concn. J. M. JACOBS

330. **Electrochemical detection of oxidising gases.** Mine Safety Appliances Co. Brit. Pat. 776,771; date appl. 11.2.54. U.S.A., date appl. 26.2.53.—A galvanic cell for the determination of the concn. of oxygen in a gas mixture containing other oxidising gases comprises a zinc anode, a carbon cathode, and an electrolyte, *e.g.*, a dil. soln. (2 to 5%) of NH_4Cl acidified with HCl or H_2SO_4 to a pH of < 2·8 (3·0), at which point no development of H at the anode is observed. The area of interaction between the polarised electrode and the electrolyte is smaller than the active area of the other electrode by at least an order of magnitude, and the smaller electrode area is in inverse proportion to the max. concn. of the oxidising component to be tested with a given electric meter. J. M. JACOBS

331. **Zone electrophoresis on sponge rubber.** H. K. Mitchell and L. A. Herzenberg (California Inst. Technol., Pasadena, U.S.A.). *Anal. Chem.*, 1957, **29** (8), 1229.—Foam-rubber sponge is a convenient and useful supporting medium for zone electrophoresis of proteins and other substances. Samples can be recovered afterwards simply by squeezing the sponges. K. A. PROCTOR

332. **Improvements in or relating to polarographs.** Cambridge Instrument Co., Ltd., and G. Jessop. Brit. Pat. 776,543; date appl. 1.7.53.—Reduction of the effect of series resistance in polarograph apparatus is obtained by supplementing the electrode voltage by a further alternating voltage, the amplitude of which is proportional to the electrode current, and which is of the same phase as the electrode current. Thus, *e.g.*, in the arrangement described in B.P. 640,768, the electrode current is amplified by a suitable amplifier, at an output of which, before rectification, a voltage which is of the same phase as, and proportional in amplitude to, the electrode current is obtained. A predetermined fraction of this voltage is superimposed on the voltage applied to the electrode. If this fraction is properly adjusted according to the value of the series resistance, the resulting total electrode current is of the same magnitude and phase as it would be if there were no series resistance. J. M. JACOBS

333. **Calibration of an integrating motor for coulometric titrations.** N. H. Furman and A. J. Fenton, jun. (Frick Chem. Lab., Univ., Princeton, N.J., U.S.A.). *Anal. Chem.*, 1957, **29** (8), 1213–1214.—Extensive calibration has shown that an empirical relationship which is linear over a range of current levels and resistances exists between the motor calibration factor and the value of the series resistor. By using this relationship, motor factors can be calculated with an accuracy of 1 to 2 parts per thousand. K. A. PROCTOR

334. **A solid state paramagnetic resonance spectrometer.** P. M. Llewellyn (Clarendon Lab., Univ. Oxford). *J. Sci. Instrum.*, 1957, **34** (6), 236–239.—A spectrometer is described which is a development of that described by Bleaney and Stevens (*Rep. Progr. Phys.*, 1953, **16**, 108) and is used in the frequency range 9000 to 10,000 Mc/s at the fixed temp. of boiling refrigerants, the lowest temp. being the triple point of hydrogen (14° K). A high sensitivity is achieved by modulating the magnetic field at 115 kc/s. The sensitivity is such that 10^{13} atoms of ^{56}Co have been detected at 20° K, and it is estimated that 2×10^{-11} mole of the free radical diphenyltrinitrophenylhydrazyl would be detectable at 290° K. N. E.

335. **Mass spectrometry applied to clinical practice and research.** K. T. Fowler and P. Hugh-Jones (Postgrad. Med. School, London). *Brit. Med. J.*, 1957, 1205–1211.—An instrument designed specifically for the study of human respiratory function is described. Four gases, in the mol.-wt. range 18 to 80, can be simultaneously and continuously determined throughout the respiratory cycle by using only 0·1% of the patient's exhaled breath. The response time is less than 0·1 sec., changes in concn. of 1% can be detected, and the instrument is transportable. H. F. W. KIRKPATRICK

336. **Oscillographic mass-spectrometric monitoring of gas chromatography.** J. C. Holmes and F. A. Morrell (Philip Morris Inc., Richmond, Virginia, U.S.A.). *Appl. Spectroscopy*, 1957, **11** (2), 86–87.—A method is described for continuously monitoring the eluate from a gas-chromatographic column with a mass spectrometer and an oscilloscope. Any sixteen-mass-unit portion of a spectrum can be displayed and the smallest change in the pattern immediately detected. The scan rate can be altered to decrease the time required for the display or to present a larger portion of the spectrum. The method is applicable to the study of reaction rate, thermal cracking, etc., and the presence of intermediate compounds formed during a reaction can be detected quickly. A commercially available mass spectrometer can be easily adapted for this purpose. K. A. PROCTOR

337. **Neutron moisture-meter for concrete.** J. Pawliw and J. W. T. Spinks (The Univ., Saskatoon, Saskatchewan, Canada). *Canad. J. Technol.*, 1957, **34** (8), 503–513.—A portable instrument for determining rapidly the total content of H_2O in a plain or lightly reinforced concrete member, without destruction, is described. Fast neutrons from a Ra-Be source (10 or 50 mC) are converted into slow neutrons by the H_2O mol. and are then determined with a BF_3 counter (2 or 4 in. long). The meter is calibrated against concrete cylinders of known H_2O content; its sensitivity is less than that of the similar soil-moisture meter because it is placed against the concrete surface. W. J. BAKER

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cycles per second	c/s
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density, relative	d or wt. per ml
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electromotive force	e.m.f.
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kilowatt	kW
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micron	μ
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relative humidity	r.h.
revolutions per minute	r.p.m.
saponification value	sap. val.
saturated calomel electrode	S.C.E.
second (time)	sec.
soluble	sol.
solution	soln.
specific gravity	sp. gr.
specific rotation	$[\alpha]_\lambda^t$
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